DESIGN OF ROTARY, TURBO-MOLECULAR AND CRYO-SORPTION PUMPING SYSTEMS FOR VACUUM LABORATORY

A Thesis Submitted to

National Institute of Technology, Rourkela

In Partial fulfillment of the requirement for the degree of

Master of Technology

in

Mechanical Engineering

By

SHIHABUDEEN P.S.
(Roll No.212ME5329)

Department of Mechanical Engineering
National Institute of Technology
Rourkela -769 008 (India)
2014

DESIGN OF ROTARY, TURBO-MOLECULAR AND CRYO-SORPTION PUMPING SYSTEMS FOR VACUUM LABORATORY

A Thesis Submitted to

National Institute of Technology, Rourkela

In Partial fulfillment of the requirement for the degree of

Master of Technology

in

Mechanical Engineering

By

SHIHABUDEEN P.S.
(Roll No.212ME5329)

Under the supervision of

Prof. SUNIL KUMAR SARANGI

Department of Mechanical Engineering
National Institute of Technology
Rourkela -769 008 (India)
2014

National Institute of Technology

Rourkela

CERTIFICATE

This is to certify that the thesis entitled "**Design of Rotary, Turbo-Molecular and Cryo-Sorption Pumping Systems for Vacuum Laboratory**" being submitted by Shihabudeen P.S. (212ME5329) for the partial fulfillment of the requirements of **Master of Technology degree** in **Cryogenic & Vacuum Technology** is a bonafide thesis work done by him under my supervision during the academic year 2013-2014 in the Department of Mechanical Engineering, National Institute of Technology Rourkela, India.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Place: NIT Rourkela
Date:

Prof. Sunil Kumar Sarangi
Department of Mechanical Engineering
National Institute of Technology, Rourkela

Acknowledgment

With great feeling and immense pleasure I would like to express my thanks and gratitude to my project supervisor Prof. Sunil Kumar Sarangi, Mechanical Engineering, NIT Rourkela, who spared a great amount of his valuable time for giving me guidance, help and encouragement over the last one year.

I express my sincere thanks to Prof. K.P.Maity, HOD, Mechanical Engineering, NIT, Rourkela for providing me the necessary facilities in the department. I am extremely grateful to Prof. R.K.Sahoo, for his help in purchasing the equipment and timely advice in various occasions.

My heartfelt thanks to Prof. V.V. Rao, IIT Kharagpur for the support and help extended while doing the Laboratory experiments.

A special thanks goes to my class mate, Trilochan Penthia, who has been working with me during the entire one year in helping to purchase and assemble the equipment.

I take this opportunity to thank all research scholars for being very helpful and especially to Mr.Rvindra Vutukuru for his cooperation and assistance.

For many technical as well as mechanical helps, I must thank workshop and lab instructors for being kind enough to help any time during this period.

Shihabudeen PS

ABSTRACT

This project deals with study, design, construction and installation of Vacuum laboratory apparatus including preparation of lab manual for Vacuum Laboratory which is being set up under Refrigeration and Cryogenic Engineering Centre in NIT Rourkela.

The Vacuum laboratory, a part of first year M.Tech (Cryogenics and Vacuum Technology) curriculum in Mechanical department, consists of twelve experiments in which six experiments will be designed and installed under this project. The proposed laboratory will help both the graduate and under graduate students of our institute to conduct different experiments under vacuum environment.

Two experiments using rotary pump [1] namely; Pumping speed measurement and conductance measurement of different piping analogy need to be set up. For which we designed a vacuum chamber [3] with suitable capacity first, decided pump down time allowable to get ultimate vacuum level of a rotary pump in accordance with the total lab hrs assigned to each sessions, and finally we calculated pumping speed [2] required for the pump. The same rotary pump set up will be used for conductance measurement too, but with different piping analogy. Conductance of each pipe is calculated using relations and are compared with experimental results in series and parallel connections.

For high vacuum application a turbo-molecular pump [4] experimental set up is designed. Pumping speed calculation as well as leak detection test may be carried out in this set up. For roughing and backing, a suitable rotary pump is fitted with this pump.

Since Mass spectroscopic leak detection [5] is most commonly used leak detection method in vacuum systems, we proposed one MSLD with turbo-molecular pump. Bill of material for the turbo pump is also prepared and handed over to department.

Oil-free vacuum atmosphere can also be created in our lab by using sorption pump [6] for medium range vacuum applications. A small capacity sorption pump's design is carried out and drawings are prepared.

Keywords: *Cryogenics, Turbo-molecular pump, Sorption Pump, Mass spectroscopy*

Contents

List of Figures

List of Tables

Introduction

Design of various vacuum system parameters like thickness of the vessel and length of test pipeline, leak rate, pumping speed etc are to be calculated as part of this project. After the equipment is designed the item lists are prepared for all experimental set up. Vacuum vessels are designed using basic hoop stress equations and it is made out of stainless steel. Piping, valves and sealing materials are readily available. Assembling of equipments are done as per the drawing prepared. Experiment has to be done and result should be verified with standard data provided by manufacturers. Also, experimental readings, tables & graphs for these experiments are documented so that they can be used as standard results for later use by students.

List of Experimental Setups

The following experimental setups to be designed under this project.

1. Study of Rotary Pump. Pumping speed measurement of Rotary Pump and Characteristic curves; Pumping Speed Vs Pressure.

2. Conductance measurement of different piping analogy.

3. Study of Turbo-molecular Pump and Characteristic curves; Pumping Speed Vs Pressure.

4. Study of Sorption Pumps and its pumping Speed measurement.

CHAPTER 1

PUMPING SPEED MEASUREMENT OF ROTARY PUMP

Aim

To design and construct a Rotary pump experimental set up to measure pumping speed.

1.1. Mechanical Pump

In this section we will discuss the theory of operation of mechanical pump and its application. Almost all vacuum application requires mechanical pump for roughing and backing operation. So mechanical pumps can well be considered as an important part of all vacuum systems. It requires less maintenance, cheap and can be operated on long run basis even without failure. Classification of mechanical pump is given as below;

1.2. Classification

Mechanical positive displacement pumps

Reciprocating pumps

- Diaphragm pump
- Piston pump

Rotary Pumps

- Sliding vane pump
- Liquid ring pump
- Rotary piston pump
- Rotary plunger pump
- Multiple vane rotary pump
- Roots pump

For our laboratory, we design and set up an oil sealed sliding vane pump.

1.3 Theory of Operation

Mechanical vacuum pumps are categorised as positive gas displacement pumps. During working the pump entrap some fixed volume of air or fluid in between a pair of blades or lobes by successive closing of outlet and inlet valves. Again the entrapped volume gets compressed eventually by further rotation of blade. Due to reduction in volume compressed gas itself push open the outlet valve or by means of a mechanical drive, through which the displaced volume

of gas escapes to atmosphere. Generally rotor blades slide inside the stator slots which cut radially and rotate simultaneously by the shaft driven an electric motor. The ratio of exit pressure (atmospheric) to the inlet pressure (system pressure) is termed as the Compression Ratio.

In principle, vacuum pumps work same as compressors, run with the inlet connected to the system where vacuum is to be created and maintained and the outlet open to atmosphere. Most commonly, compressors and vacuum pumps are identical machines. But manufacturers strongly recommend that the same machine not be used interchangeably. The heavy loads during compression may damage it. Selection of pump should be based on: range of vacuum produced, air removal rate, leak in rate and power requirement.

1.4. Rotary Vane Mechanical Vacuum Pumps

It is a simple and most commonly used positive displacement pump. Fig shows the internal details of a rotary pump. It has a rotor and a stator. Stator houses a cylindrical rotor which has two radially machined slots opposite to each other. Two spring loaded vanes are inserted in these slots which tightly rub the stator casing when it rotates. Also the rotor makes a tight contact with stator at its top. Two holes are drilled on each sides of the stator from its vertical axis to which inlet and exhaust ports are connected.

Internal mating surfaces are machined with high precision and have micron level clearance. Stator –rotor assembly is immersed in oil which acts as coolant as well as lubricant. Always exhaust gas passes through the oil and escapes to atmosphere.

Rotary pumps typically connected to an electric motor which drives the rotor of the pump. Centripetal causes the vanes in the spinning rotor to force them against the inner contact surface of the stator. Sometimes springs can be used to improve this action. As each vane passes the inlet port, volume between the stator and rotor increases and a low pressure is created inside the confined space which sucks the air from the system through the inlet port. Sucking of air continues till the vane reaches bottom position. Another half rotation of the stator does not suck the air but compress the entrapped air until the pressure is sufficient to open exhaust valve. Exhaust gas escape out through the oil to atmosphere. Since the two vanes operate simultaneously with the rotor, suction as well as compression take place in each rotation.

Rotary pumps are available in single or double stage design. Single stage pumps are simpler, having only one stator- rotor assembly, and are cheap. In two stage pump, the inlet port of the second stage is connected in series with the exit port of the first stage.

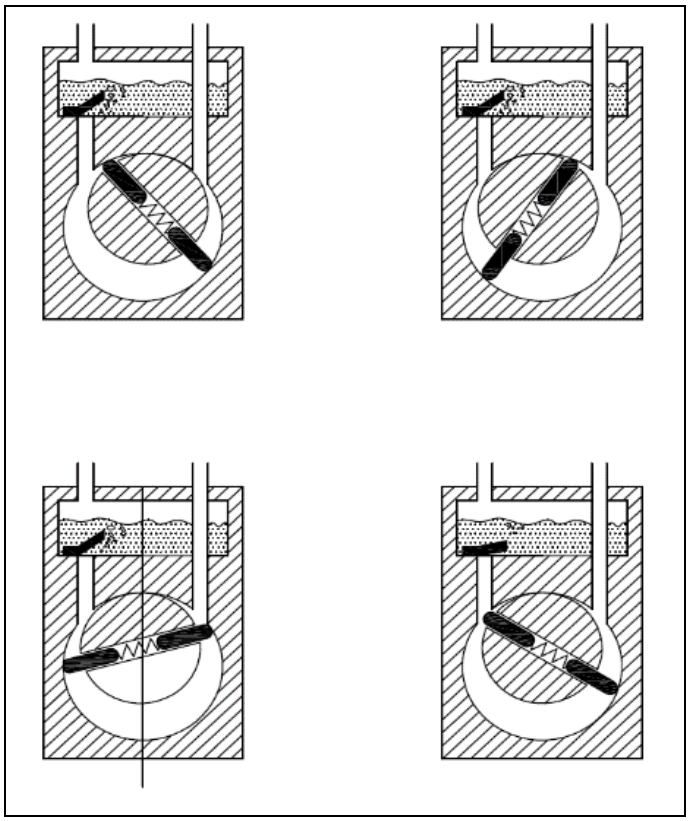

Fig 1.1. Constructional Details of Rotary Pump

1.5. Design

1.5.1. Vacuum Vessel Design Parameters

Designing a vacuum chamber means defining the boundary conditions ie deciding the external loads (atmospheric pressure and axial load due to accessories fitting on the vessel) choosing the material, designing the ports, deciding the welding methods, manufacturing and assembling these parts together and putting them under vacuum.

Cylindrical tubes are the most commonly shape for vacuum chambers. It is most stable and its analysis is simple compared to other shapes. Due to this reasons we select cylindrical shape for our vacuum lab purposes.

The differential pressure to be considered on vacuum chamber is obviously atmospheric pressure ie; one bar during operation but it is recommended to give a good factor of safety to prevent it from damage caused by over-pressure while purging with liquid nitrogen or to provide a safety valve.

Stresses

Stresses generated by external atmospheric pressure should remain in the elastic range. The equivalent stress developed due to circumferential and axial stresses (according to the Von Mises or Tresca criterion for example) should not exceed the elastic limit anywhere in the structure.

In our design, stress developed in the chamber mainly due to an external pressure results, membrane undergo compressive stress. Under these conditions, the membrane strain energy can be converted to bending strain energy, leading to an instability and a bifurcation point on the behavioural curve, a potential buckling. Buckling is a non-linear phenomenon and it could be strongly influenced by the defects inherent to the manufacturing.

Vacuum vessel is subjected to external pressure and its magnitude is equal to atmospheric pressure.

Circumferential stress developed under external pressure *p* is:

$$\sigma_\theta = \frac{pR}{t} \quad (2.1)$$

Where, *R* and *t* are radius and thickness of the circular tube respectively,

In this design tube is closed and will subjected to an axial force *F*, the axial stress is

$$\sigma_x = \frac{pR}{2t} + \frac{F}{2\pi Rt} \quad (2.2)$$

And the Von Mises equivalent stress to be compared to the material maximum allowable stress is

$$\sigma_e = \frac{1}{2}\left[3\left(\frac{pR}{t}\right)^2 + \left(\frac{F}{\pi Rt}\right)^2\right]^{1/2} \quad (2.3)$$

The buckling pressure can also be calculated using analytical formulas, depending upon the geometric size of the tube and the Young's modulus of the material.

$$P_{cr} = \frac{0.25E}{1-v^2}\left(\frac{t}{R}\right)^3 \quad (2.4)$$

Where, *v* being Poisson ratio and $(1 - v^2)$ can be taken as 0.9.

A common rule of thumb always follows is that the thickness of a stainless-steel circular tube should be at least one hundredth of its diameter (safety factor included).

1.5.2. Evacuation of a chamber in rough vacuum region

In rough vacuum range the effective pumping speed Seff, of pumping system is dependent only on the target pressure p, total volume V of the container, and the pump-down time t.

In this case the required pressure, $p_d >> p_{end}$, assuming a constant pumping speed S_{eff}, the rate of change of pressure with respect to time is given by;

$$\frac{-dp}{dt} = \frac{Seff}{V}.p \tag{2.5}$$

p_d is required or desired pressure and p_{end} is ultimate pressure that can be attainable.

Pumping starts at 1013 mbar with time t = 0, the S_{eff} is calculated which depend only on the time t from equation (1) as follows:

$$\int_{1013}^{p} \frac{dp}{p} = -\frac{Seff}{V}.t \tag{2.6}$$

$$Ln\frac{p_d}{1013} = -\frac{Seff}{V}.t$$

$$S_{eff} = \frac{V}{t}.\ Ln\frac{1013}{p_d} \tag{2.7}$$

So time required to pump down, $t = \frac{V}{Seff}.\ Ln\frac{1013}{p_d}$ (2.8)

$$t = \tau^* \sigma \tag{2.9}$$

The equation contains two variables; t and S_{eff} whereas V and p_d are constants. For designing a vacuum system we can either pump-down time or effective pumping speed. In almost all cases required vacuum level and volume to be evacuated is fixed.

Or in other words $\frac{V}{Seff}$ is known as time constant, τ which means time required to pump down in rough vacuum region depends only on the required pressure. $Ln\frac{1013}{p_d}$ is a dimensionless factor.

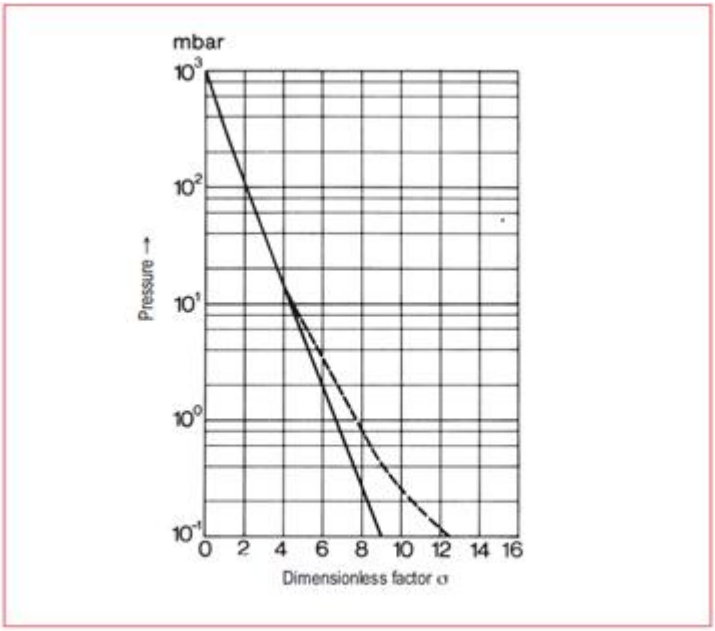

Fig. 1.2: Dependency of dimensionless factor σ on Pressure

These calculations are valid only when the required pressure is several orders of magnitude higher than the ultimate pressure.

1.5.3. Evacuation of a chamber in the medium vacuum region

In the rough vacuum region, the time required to pumping process depends on the volume of the vessel. In the high and ultrahigh vacuum regions, however, the gas evolution and leak-in rate from the walls plays a significant role. Whereas in medium vacuum region, the time taken is influenced by volume as well as gas evolution and leak-in rate. Also in the medium vacuum system, maintained by rotary pumps, the ultimate pressure p_{end} is cannot be negligible. If the quantity of gas entering the chamber is known to be at a rate Q (in millibars litre per second) from gas evolution from the walls and leakage,

If Q is the gas entering the chamber by leakage and gas evolution from the chamber walls, the rate of change of pressure with respect to time is given by;

$$\frac{-dp}{dt} = \frac{\text{Seff}}{\text{V}} \cdot \text{p}$$

Then differential equation for present case becomes;

$$\frac{dp}{dt} = -\frac{\text{Seff}(p_d - p_{end}) - Q}{\text{V}} \quad (2.10)$$

On integrating

$$\text{t} = \frac{\text{V}}{\text{t}} ln \frac{(p_0 - p_{end}) - Q/S_{eff}}{(p_d - p_{end}) - Q/S_{eff}} \quad (2.11)$$

Where p_0, p_d and p_{end} are the pressure at the beginning of the pumping process, the desired final pressure and ultimate respectively.

Using this equation we cannot find out a definite solution for effective pumping speed S_{eff}, so for practical purposes the following methods are mostly adopted and we generally go for a pump with high pumping speed to eliminate the possible difficulty to maintain vacuum with leakage Q:

a) The pumping speed is calculated using equation without considering gas evolution for a vacuum range which is not far from rough vacuum.

b) The gas evolution rate and leak rate are found. Then choose a pump with suitable pumping speed but in no way the sum of gas evolution and leak rate is greater than effective pumping speed. Effective pumping speed must always be about ten times higher than the total of gas evolution and leakage.

1.5.4. How to determine pump size

The following steps are followed while deciding a pump size for vacuum system.

- First calculate the volume of chamber and decide the required vacuum level.
- Calculate the total conductance of piping so that we could find out the effective pumping speed which is available at the evacuation port of the chamber.
- Find out the leakage rate and gas evolution from all surfaces of the system.
- Decide the pump down time we need to allow, within which we can bring down the system to a required pressure level.
- Now we can calculate the effective pumping speed required to reduce the pressure within the certain time either by using formulae or nomogram.

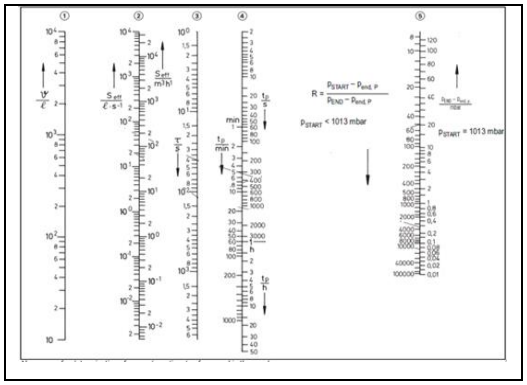

Fig 1.3. Nomogram

Pumping Speed Requirement

$S_{eff} = \frac{Q_l}{P_{end}}$, $\rightarrow S_{eff} = \frac{5*10^3}{8*10^{-3}} = 2.25\ m^3/h$, $Q_l = 5*10^{-3}$ mbar l/s, $P_{end} = 8*10^{-3}$ mbar

$\frac{1}{S_{eff}} = \frac{1}{S_{max}} + \frac{1}{C} \rightarrow \frac{1}{S_{max}} = \frac{1}{0.625} - \frac{1}{1.9}$ $\rightarrow S_{max}$ = 3.86 m3/h, C = Conductance of DN 25 KF (1 m) Flexible Hose from Table 2.2 considering molecular flow (least value for safe design).

For series conductance measurement of DN 40+25 KF,

$\rightarrow \frac{1}{S_{max}} = \frac{1}{0.625} - \frac{1}{1.531}$ $\frac{1}{C_T} = \frac{1}{8} + \frac{1}{1.9}$, $C_T \rightarrow 1.53$ l/s

$S_{max} = 3.78\ m^3/h$

So the pump should at least have a capacity of 3.78 m^3/s to attain $8*10^{-3}$ mbar with DN 40+25 KF pipe combination.

∴ We select a pump with a capacity of 6 m^3/h.

1.6. Vacuum Vessel

Volume required for the vessel, V= 25 Ltr

$V = \frac{\pi D^2}{4} * L$

Length of vessel, L= $\frac{0.025*4}{\pi*300^2}$ = 355 mm, but we consider L =360 mm, Take D= 300 mm

1.6.1. Thickness

(a) Using eqn (2.1)

Where atmospheric pressure, p= 101.325 kPa, Yield Strength of SS 304, σ_y = 205 MPa, Permissible Hoop Stress, σ_θ (safety factor as 3) = $\frac{\sigma_y}{3}$ = 205/3= 70 MPa, Radius, R= 0.15m

$\sigma_\theta = \frac{pR}{t}$ $\rightarrow t = \frac{pR}{\sigma_\theta}$ $\rightarrow t = \frac{101.325*10^3*0.15}{70*10^6}$ $\rightarrow$ t = 0.217 mm,

(b)Using eqn (2.2)

$\sigma_x = \frac{pR}{2t} + \frac{F}{2\pi Rt}$, Axial force F is calculated as $p * \frac{\pi {D_0}^2}{4}$ $\rightarrow$ F = $101.325 * 10^3 * \frac{\pi*300^2}{4}$

= 7158 N

Top cover plate effective diameter, D_0 = 300 mm

Permissible axial Stress, $\sigma_x = \frac{\sigma_y}{3}$ = 205/3= 70 MPa

$70*10^6 = \frac{101.325*10^3*0.15}{2t} + \frac{7158}{2\pi*0.15t}$ $\rightarrow$ t = 0.22mm

(c)Using eqn(2.3)

$\sigma_e = \frac{1}{2}\left[3\left(\frac{pR}{t}\right)^2 + \left(\frac{F}{\pi Rt}\right)^2\right]^{1/2} \rightarrow 70*10^6 = \frac{1}{2}\left[3\left(\frac{101.325*10^3*0.15}{t}\right)^2 + \left(\frac{9743}{\pi*0.15*t}\right)^2\right]^{1/2}$

Permissible equivalent Stress, $\sigma_e = \frac{\sigma_y}{3}$ = 205/3= 70 MPa $\rightarrow$ t = 0.239 mm

(d)Using eqn(2.4)

$P_{cr} = \frac{0.25E}{1-\nu^2}\left(\frac{t}{R}\right)^3$ Taken P_{cr} = 101.325 kPa, $1-\nu^2$=0.9 and E=200 GPa

$101.325*10^3 = \frac{0.25*200*10^9}{0.9}\left(\frac{t}{0.15}\right)^3$ $\rightarrow$ t = 1.88 mm

(d)Using Reference [7]

Cylinder

1. $\frac{D_0}{t} \leq 105,\ t \geq \frac{324}{105}$ $\rightarrow$ t = 3.08 mm, Where D_0 = Outer Diameter of pipe
2. $\frac{L_c}{D_0} \leq 11.5,\ L_c \leq 11.5 * 300 \rightarrow L_c \leq 3450\ mm$.Since cylinder length, 360 mm, is within the critical range, the design is safe.

End plates

1. $\frac{D_i}{t_c} \leq 89, \quad t_c \geq \frac{300}{89}, \rightarrow$ t = 3.37 mm.
2. Using Roark's formula

Roark's formula for end plate (flat plates supported at the edges & uniformly loaded)

Maximum stress; σ max = $\frac{3W(3m+1)}{8\pi m t_c^2}$, μ=0.3 ; so, m=1/(0.3) → m=3.33

Where; m=(1/μ), t_c= end plate thickness, r_c= End plate radius and

W=total uniformly applied load, is given by;

$W=P*\pi r_c^2$ → $W=101.325*10^3*\pi*0.172^2$, W=9412N, Where P=101.325kPa

r_c= Cylinder outer radius, R_0 + 10 mm(for providing a collar for claw clamps)

∴ r_c= 172 mm

$\sigma_{max}= \frac{3W(3m+1)}{8\pi m t_c^2} \rightarrow 205*10^6 = \frac{3*9412(3*3.33+1)}{8\pi*3.33*t_c^2}$ → t= 4.05 mm, taken σ_{max}=205MPa

1.6.2. Validity check for End Plate thickness

Young's modulus of elasticity, E=200 GPa

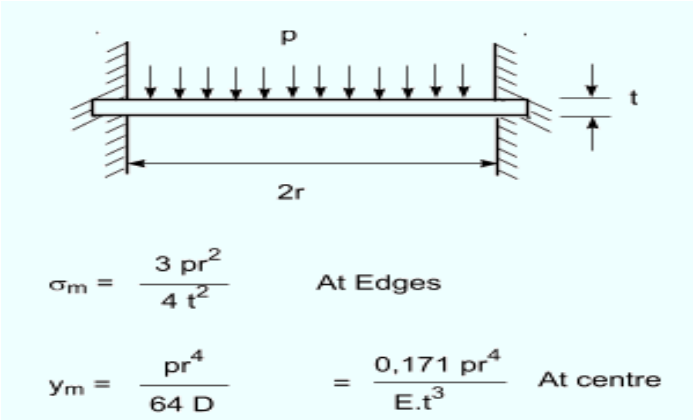

Fig 1.4. Deflection of circular end plate, uniform load, edges clamped

Maximum Deflection at centre, $y_{max}= \frac{0.171pr_c^4}{Et_c^3} \rightarrow \frac{3*101.3*10^3*0.172^4}{200*10^9*0.00405^3}$, y_{max} = 1.14mm

Now as y_{max} =1.14 mm < t_c/2 ∴Roark's formula is valid in this case &design is safe

Considering factor of safety =2.3

t min =4.05*2.3 $\rightarrow$ t min=9.315mm so, we take t=10mm

By considering the values of above calculation, tube and end plate thickness are decided.

Tube Specification	304L 12" SCHEDULE 5 SS WELDED OR SEAMLESS PIPE
OD	323.85 mm
Thickness	3.96 mm
Length	360 mm
End Plate Thickness	10mm

Table 1.1 Vacuum Vessel Pipe details

1.7. Rotary Pump Experimental Setup

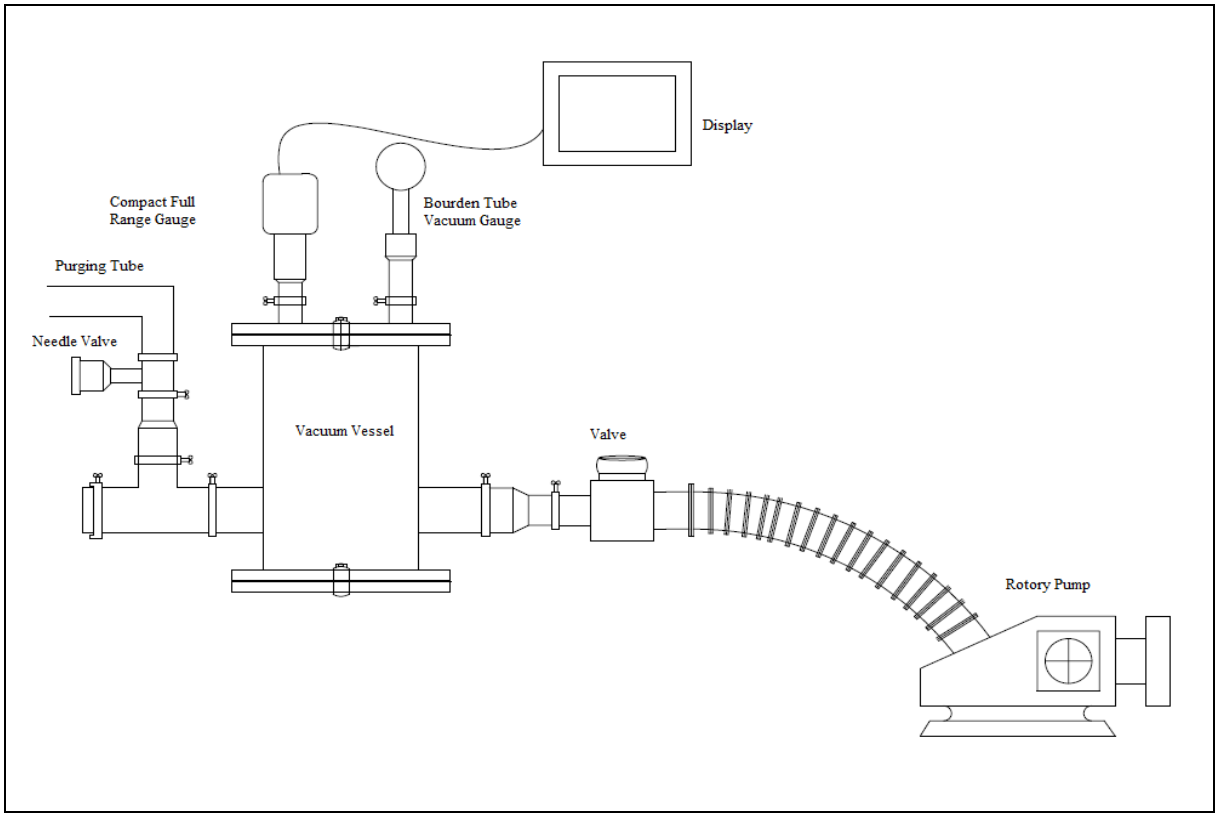

Fig 1.5. Rotary Pump Experimental Setup

1.8. Item List- Rotary Pump Set up

S.No	DESCRIPTION	SPECIFICATION	QTY
1	Vacuum Vessel	20 Ltr,Ports-2X DN 25 ISO-KF Side, 2XDN 25 ISO-KF Top	1
2	Rotary Pump- Single Stage	6 m3/Hr,100 L/min	1
3	Pfeiffer Compact Full Range Gauge	PKR 251	1
4	Dual Channel Control Unit	TPG 262	1
5	Connection Cable for TPG 262	3 mtr Cable	1
6	Bourden Tube Vacuum Gauge(1013-50 mbar)	DN 25 ISO-KF(Compatible with hinged clamps)	1
7	Needle Valve(Manual)	DN 16 ISO-KF	1
8	Right Angle Valve(Manual)	DN 25 ISO-KF	1
9	Reducing Tee	DN25-16 ISO- KF	1
10	Hinged clamp	40 KF	2
11	Hinged clamp	25 KF	8
12	Hinged Clamp	16 KF	3
13	Centering Ring	DN 40 KF	2
14	Centering Ring	DN 25 KF	7
15	Centering Ring	DN 16 KF	3
16	Flexible Hose	DN 25 KF	1
17	Flexible Hose	DN 16 KF	1
18	Flexible Hose	DN 40 KF	1
19	Conical	DN25KF-DN16 ISO-KF	1
20	Conical	DN40KF-DN25 ISO-KF	2
21	Conical	DN40KF-DN16 ISO-KF	1
22	Blank Flange	DN25KF	1

Table 1.2 Item List-Rotary Pump

1.9. Pumping Speed by Constant Volume Method:

For calculating pumping speed, we need a rotary mechanical pump, a vacuum chamber, and vacuum gauges with a range 1013- 0.003 mbar.

Setup for constant volume method is shown in fig. Connections are made as per the drawing using minimum connecting line. Start the exercise with all valves closed and the vessel being at atmospheric pressure. Switch on the mechanical pump, and aft allow it to warm up, open the valve to the vacuum vessel and start recording time required to achieve a pressure of 100 mbar using the gauge mounted on the vessel. Until we are confident repeat this measurement to ensure consistency of readings. Now note the time required to pump from 100 mbar to 10 mbar. And step by step we finally, record the time required to pump from 10 mbar to 1mbar.

1.9.1. Procedure:

1. Connections are made tight to get a leak free vacuum system.
2. Ensure the proper gauges are fitted on the system to measure the full range vacuum of rotary pump ie; Bourden tube vacuum gauge (1013-100 mbar range) and combination gauge (100- $1*10^{-2}$ mbar range).
3. Open the needle valve and bring the system pressure to atmospheric level.
4. Close the needle valve and pump out the system to 100 mbar.
5. Again admits LN2 gas into system by opening the needle valve.
6. When it comes to atmospheric pressure close the needle valve and switch on the pump provided the isolation valve is closed.
7. After starting the pump open the isolation valve and simultaneously start the stop watch.
8. Take the vacuum gauge reading after a particular interval of time.
9. Continue taking reading until the system reaches its specified ultimate pressure or its steady state.
10. Close the isolation valve and switch off the pump.
11. Calculate the pumping speed for each discretised pressure range.
12. Plot the graphs.

1.9.2. Tabulation

SNo	t (in S)	dt	p(mbar)	S_p (L/s)	P_{avg}

$$S_p = 2.3\left[\left(\frac{V}{t_2-t_1}\right)\log\left(\frac{p_1}{p_2}\right)\right]$$

Now we can plot the graph between Pumping speeds Vs Time.

The setup is designed and installed and pumping speed experiment has been done. Following is the graph obtained from the experiment.

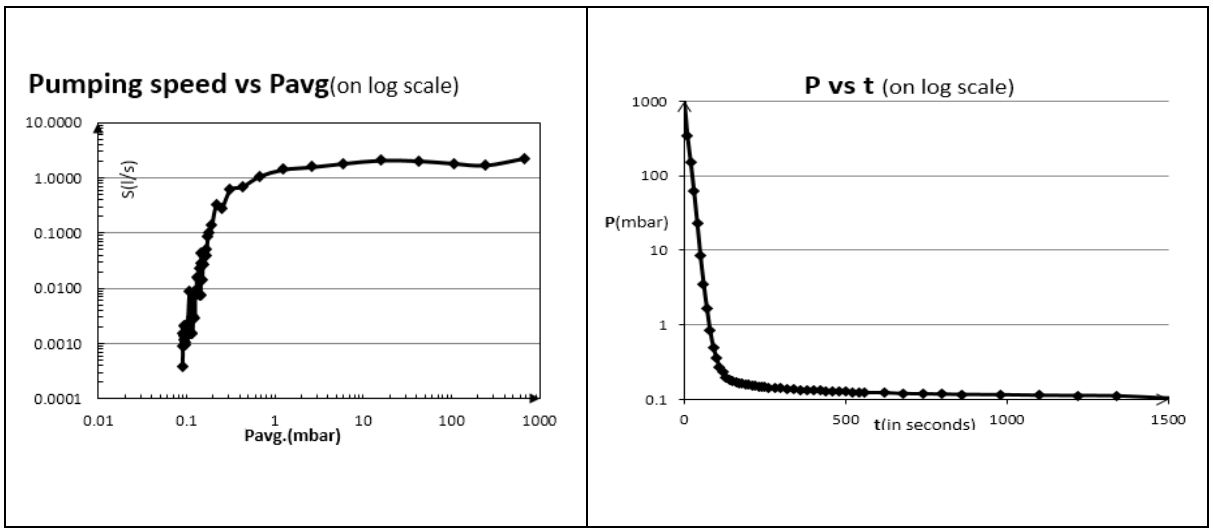

Fig 1.6. Pumping Speed Characteristics

CHAPTER 2

CONDUCTANCE MEASUREMENT

Aim

To design and construct an experimental set up to measure Conductance of different piping.

2.1 Conductance in a Vacuum System:

Flow in vacuum system depends on pressure drop as well as conductance. It also depends on diameter and length of connecting tube. Conductance elements in vacuum system like pipes, tees, cross, bend, elbow, valves vessels etc. offers resistance against gas motion.

This resistance is a function pressure difference and geometry of the conductance element.

$$Z = \frac{P_1 - P_2}{Q} \tag{2.1}$$

Z= resistance [sec/litre]

P= pressure [Torr]

Q=flow rate [Torr-L/s]

Mass flow rate, usually called throughput and pressure drop can be related by a term called conductance. Or in other words inverse of resistance is known as conductance.

$$C = \frac{1}{Z} = \frac{Q}{\Delta P} \tag{2.1a}$$

Q corresponds to the current, ΔP to the voltage and C to the electrical conductance like in electrical circuits. Since the equation is analogous to electrical circuit, it is known as "Ohm's Law of Vacuum Technology"

2.2. Configuration of conductance:

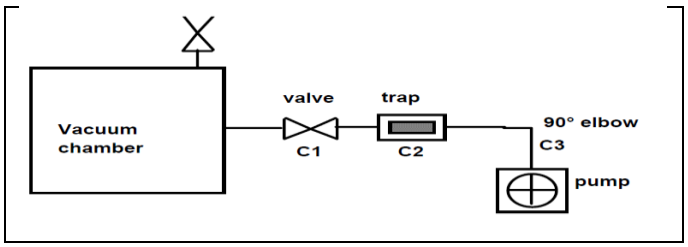

Fig.2.1.Series Connection

$$\frac{1}{C_T} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \ldots = \sum \frac{1}{C_i} \tag{2.2}$$

C_T = Total Conductance

C1, C2, C3 are individual conductance connected in series

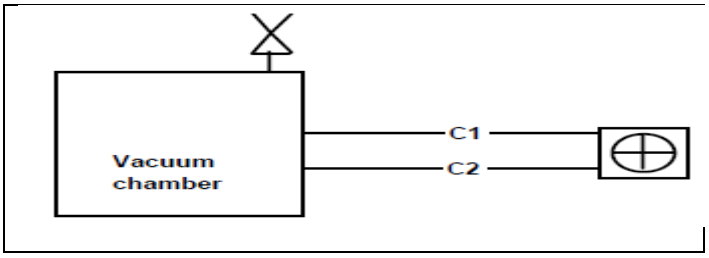

Fig.2.2.Parallel Connection

$$C = C_1 + C_2 + ... = \sum C_i \quad (2.3)$$

C_T = Total Conductance

C1, C2, C3 are individual conductance connected in series

The available or effective pumping speed of vacuum pump with a maximum speed of S_0 connected to a system by piping having total conductance of C_T is

$$\frac{1}{S_{eff}} = \frac{1}{S_{max}} + \frac{1}{C_T} \quad (2.4)$$

2.3. Conductance Calculation in different flow regime for air [1]

2.3.1. Viscous Flow: Mean free path is approximately equal to the diameter of the conductance tube; the throughput, Qv, for dry air is given by:

$$Q_v = \frac{3000\ \bar{P} D^4}{L} (P_1 - P_2) \left[\frac{\text{Torr-litres}}{\text{Sec}}\right] \quad (2.5)$$

P= average pressure = (P1+P2)/2

D= tube diameter [inches]

L= tube length [inches]

$$C_v = \frac{Q_v}{(P_1 - P_2)}, \qquad C_v = \frac{3000\ \bar{P} D^4}{L} \frac{\text{litres}}{\text{Sec}} \quad (2.6)$$

2.3.2. Molecular Flow: Mean free path is much larger than the diameter of the pipe, the throughput, Q and conductance, Cm are given by:

$$Q_m = \frac{80 D^3}{L} (P_1 - P_2) \quad (2.7)$$

$$C_m = \frac{Q_m}{(P_1 - P_2)}, \qquad C_m = \frac{80 D^3}{L} \frac{\text{litres}}{\text{Sec}} \quad (2.8)$$

2.4. Conductance values for other elements [8]

Often vacuum line contains elbows and valves, conductance of them can be found by calculating their effective length l_{eff}. The method is given below:

$$l_{eff} = l_{axial} + 1.33 * \frac{\theta}{180^{\circ}} * d \quad (2.9)$$

Where

l_{axial} : axial length of the line (in cm)

l_{eff} : Effective length of the line (in cm)

d : Inside diameter of the line (in cm)

θ : Angle of the elbow (degrees of angle)

2.5. Conductance of High Vacuum Lines (< 10^{-3} Torr) [2]

For practical purposes the flow is assumed molecular when the product of average pressure (Torr) and pipe dimension (cm) is less than 0.015. (PD<0.015)

Conductance of long circular pipe, $C_m = \frac{KD^3}{L} \left[\frac{\text{litres}}{\text{Sec}}\right]$ (2.10)

Where D & L are diameter and length of the pipe in cm.

$K = \sqrt{\left[\left(\frac{28.7}{M}\right)\left(\frac{T}{293}\right)\right]}$, M = molecular weight, K= Temperature in Kelvin (2.10a)

This formula is independent of type and temperature of the gas being pumped.

2.6. Conductance of Rough Vacuum Lines (< 10^{-3} Torr) [2]

The flow may be considered viscous if PD > 0.5. Where pressure is in Torr and D in cm.

$$C_v = 3.3*10^{-2} \left(\frac{D^4 P}{\mu L}\right) \quad (2.10)$$

μ is the viscosity in poise.

The flow will be transitional, if (0.015< PD < 0.5), and conductance is given by;

$$C_v = 3.3*10^{-2} \left(\frac{D^4 P}{\mu L}\right) + \frac{10D^3}{L} \quad (2.11)$$

The first term is due to viscous flow and second term for molecular flow.

Conductance Calculation.(0.015< PD < 0.5) Torr-cm, or (10^{-2} < PD < $6 \cdot 10^{-1}$) mbar · cm.

2.7. Average Pressure Limit of Piping for different flow regime

Element	Viscous, P (mbar)	Transition	Molecular
Flexible Hose DN 40 KF(4 cm)	1013 – 0.15	0.15 – 0.0025	Less than 0.0025
Flexible Hose DN 25 KF(25)	1013 – 0.24	0.24 – 0.004	Less than 0.004
Flexible Hose DN 16 KF(16)	1013 – 0.375	0.375 – 0.006	Less than 0.006

Table 2.1 Average Pressure Limit of piping for different flow regime

The Rotary pump experimental set up is so designed to achieve a vacuum pressure of $6*10^{-2}$ mbar. For calculating pump down time it is necessary to calculate conductance and effective pumping speed of the system.

Element	C_v- (1 mbar) in L/s		C_m in L/s	
	L=50 cm	L=100 cm	L=50 cm	L=100 cm
Flexible Hose DN 40 KF(4 cm)	700	360	15.8	8
Flexible Hose DN 25 KF(25)	100	50	3.8	1.9
Flexible Hose DN 16 KF(16)	16	8	1	0.5

Table 2.2 Conductance of piping

2.8. Experimental Set up

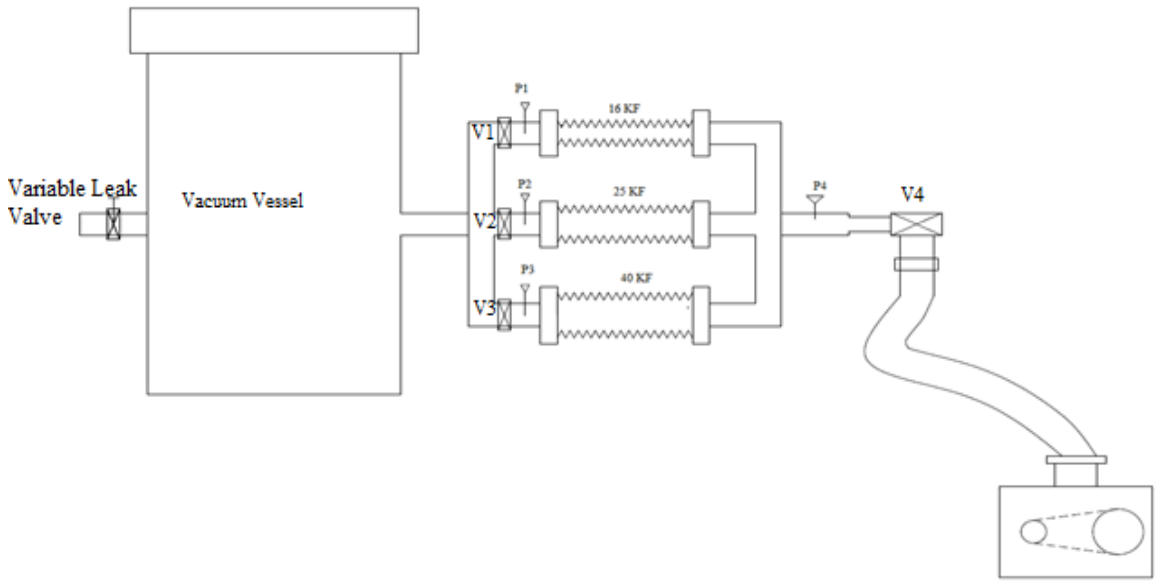

Fig 2.3. Conductance Measurement System

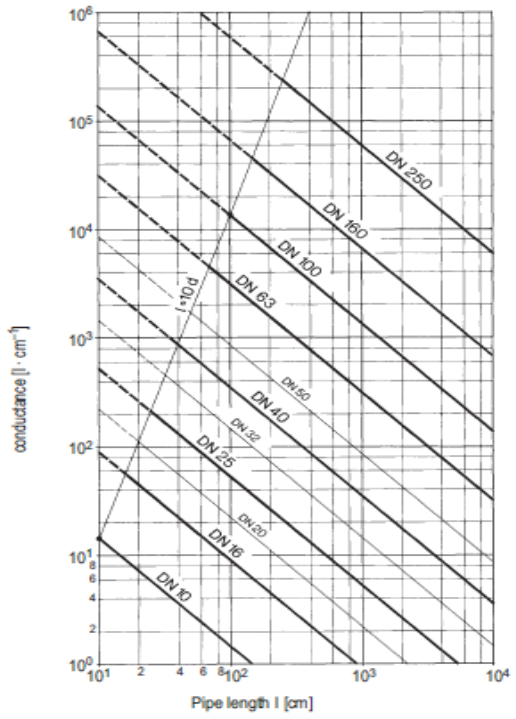

Fig 2.4 Conductance of piping for viscous flow (p=1 mbar) Reference [8] Air

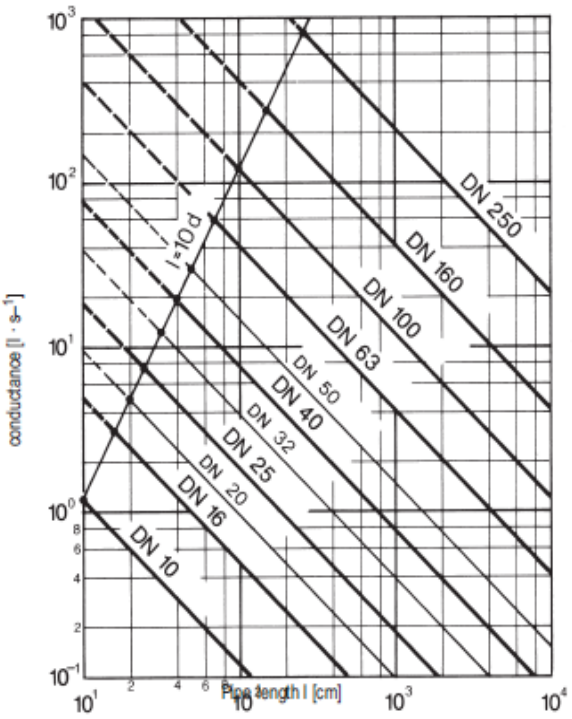

Fig 2.5 Conductance of piping for molecular flow Reference [8] Air

2.9. Procedure:

1. Isolate the particular element to be studied using valves V1, V2 and V3.
2. Admit dry air in to vacuum vessel using calibrated variable leak valve at a known mass flow rate.
3. Measure the pressure gradient across the pipe with calibrated gauges.
4. $\frac{Q}{\Delta p}$ Gives the conductance (C) of the element at the average pressure given by $\frac{p_1+p_2}{2}$.
5. Repeat the experiment at various average pressure.
6. Plot conductance versus pressure for a particular pipe.
7. Check the validity of expression $\frac{1}{S_{eff}} = \frac{1}{S_{max}} + \frac{1}{C_T}$ by measuring S_{eff} and S_{max} through pump down characteristics.
8. Repeat the above experiment for different tubes. Plot conductance of a pipe as a function of diameter at the same average pressure.
9. Measure effective conductance of parallel and series combination and check the validity of theoretical expression.

2.9.1. Tabulation

No.	t (s)	dt	P_1 (mbar)	P_2(mbar)	Pav(mbar)	Seff (l/s)	Ceff (l/s)

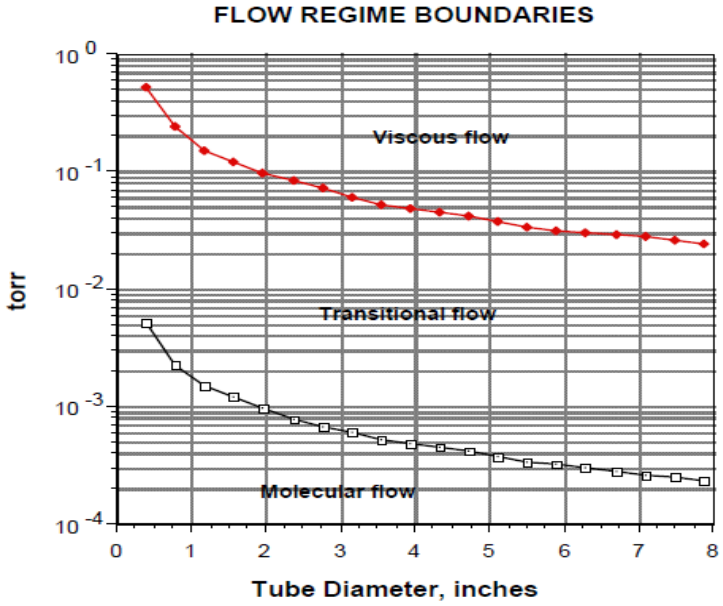

Fig 2.6 Dependency of Diameter and Pressure on Type Flow

CHAPTER 3

TURBOMOLECULAR PUMP

Aim

To design and construct a Turbo-molecular pumping station to measure pumping speed.

3.1. Principle of Operation

The turbo-molecular pump is just like a turbine with blades. It works on the principle of momentum transfer. With a rapidly rotating rotor the gas molecules at their initially non-directed thermal motion is converted to a directed motion. Hence, the pumping is achieved by directing the gas molecules from the inlet flange to the exhaust port where a backing pump has already been connected. Generally this pump is used to evacuate the chamber which is already in a vacuum range, below 10^{-3} mbar which coming under molecular flow range**.** In molecular flow regime, since the mean free path of the gas molecules is greater than the spacing between rotor and stator blades, molecules that being collide with the blades get adsorbed with rotor blades and after sometime on continuing the rotation of the rotor the thermal molecular speed is increased by absorbing momentum from rotor blades. Due to this increased momentum, molecules get detached from the preceding rotor blades and get attached to next stator blade. This process will continue until the molecules reach exhaust port.

To ensure the continuous pumping momentum that is transferred by rotor blades is not lost due to molecular collisions, molecular flow must be maintained in the pump. Mean free path must always greater than the rotor-stator blade spacing. Consequently, the molecules come in contact primarily with the rotor blades other than the molecules themselves.

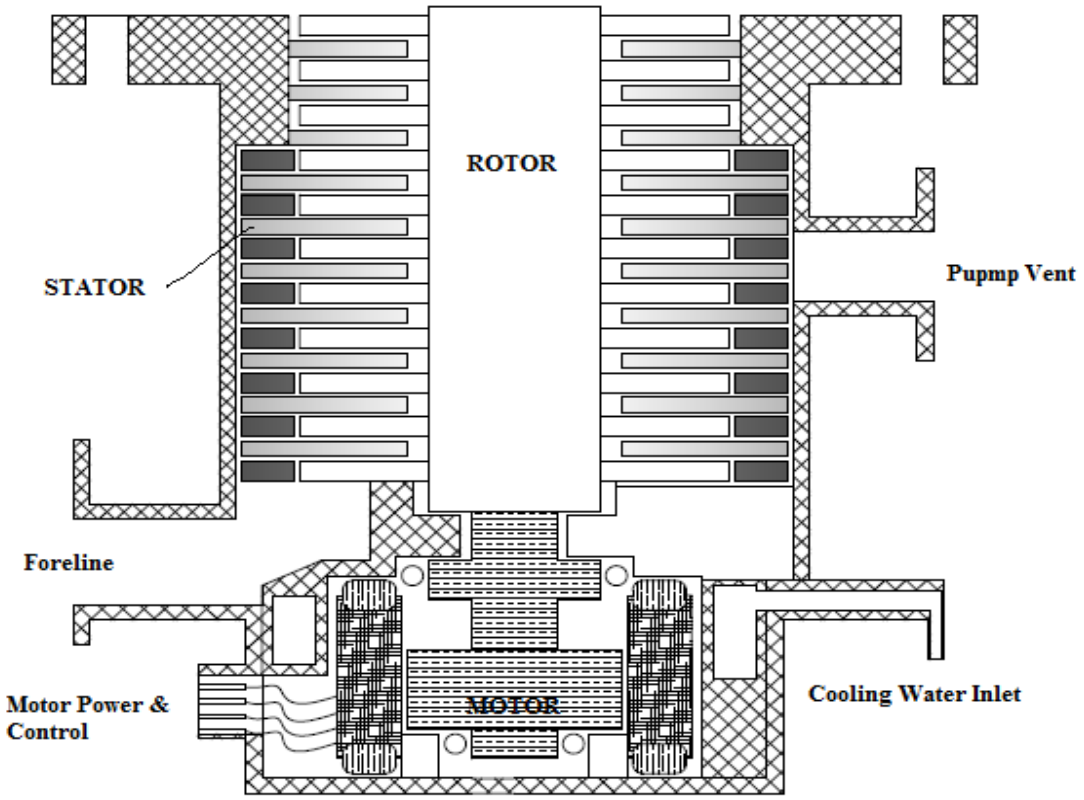

Fig 3.1 Cross Sectional View of Turbomolecular Pump

3.2. Operational Aspects of Turbo-molecular Pumps

In the molecular flow range when pressure below 10^{-3} mbar the mean free path is larger than the clearance between stator – rotor assembly. Therefore the molecules first come in contact with rotor blades rather than with molecules each other. This is the reason why this pump works efficiently within molecular flow region. Whereas when pressure exceeds the cross over limit, i.e. in laminar flow range at pressure over 10^{-1} mbar intermolecular collisions will be predominant. Due to this reason turbo-molecular pump is incapable of evacuating a system which is kept at atmospheric pressure. A fore vacuum pump is always fitted with turbo-molecular pump to bring down the pressure from atmospheric to crossover pressure. Once the system pressure is reached crossover pressure, then we can operate turbo pump for further pump down process. Its speed varies from 30,000- 90,000 rpm

Crossover pressure of turbo-molecular pumps is around 1 Torr. This is a factor of ten time higher pressure than the maximum designed crossover pressure for most oil vapour diffusion pumps (100 mTorr). At pressures above 1 Torr the turbo pump blades will be slowed by collisions with gas molecules such that the motor will overload and the rotational velocity of

the rotor will decrease to a speed that is ineffective for pumping gas. Unlike diffusion pumps, turbo pumps do have moving parts that can cause vibration which may adversely affect some precision instruments including scanning electron microscopes and surface science probes. A 60 or 120 Hz vibration typically is caused by a mechanical backing pump, while high frequency vibration is due to imbalances in the turbo pump rotor. Most vacuum applications are insensitive to this minute amount of vibration, but if vibration must be held to a minimum, and the pumping characteristics of a turbo pump are desired, a magnetically levitated rotor design may provide the solution. In this type of turbo pump conventional (but oil free) bearings are only used on start-up and shut-down of the turbo. During normal operation the rotor is suspended above the bearings by well-matched sets of strong magnets, virtually eliminating all mechanical vibration. Magnetically levitated turbo pumps are designed to operate for long periods of time with very few interruptions. Each time a magnetically levitated turbo pump is started or stopped, the oil-free mechanical bearings suffer wear and eventually will require replacement. Beyond reduction of vibration, the magnetically levitated rotor design turbo offer the option of mounting in any orientation, as there is no oil sump as in most conventional turbo pumps.

3.3. Applications for Turbo-molecular Pumps

Turbo-molecular pumps are used majorly in industries like semiconductor equipment manufacturing, thin film deposition and leak detector manufacturing. Sputter deposition methods in coating industries based on flow of a process gas, usually at pressures of 3 to 50 milliTorr are often conducted with throttled turbo-molecular pumps because of its high pumping speed characteristics. Argon gas can be pumped effectively by turbo pumps. Another application is in leak detection. Modern leak detectors for vacuum fields also often use turbo pumps as the high vacuum pump. Another desirable characteristic of turbo pumps for leak detector application is the relatively high pumping speed for atmospheric gases (oxygen, nitrogen, and carbon dioxide) compared with that for the light gas, helium. In most instances helium is used for leak detection due to its small molecular size, rarity in the atmosphere, and low toxicity. Now a days almost 90% of high vacuum applications are fitted with turbo-molecular pumps replacing oil based diffusion pump.

3.4. Characteristics

3.4.1. Gas loads

Throughput; the gas flow transported by a pump is given by;

$q_{pv} = S*p = \frac{dV}{dt} * P,$ (3.1)

This is also known as gas load on the pump .Gas load increases with pressure and when the pressure reaches critical condition, pumping speed of turbo-molecular pump declines and finally reaches zero. The maximum possible gas load of the pump depends upon the type of cooling and the nature of gas. Since the heavy noble gases having low specific heat, only little amount of heat can be dissipated to surrounding. Thus it may cause to heat up of rotor- stator blades unless we provide effective cooling system. In such condition we should regularly monitor the rotor temperature and control the RPM accordingly to ensure the safety of the pump.

3.4.2. Critical backing pressure

Critical backing pressure is the maximum pressure provided by backing pump on the backing-vacuum side of the turbo-molecular pump beyond which the pump's compression decreases. The maximum critical backing pressure is always specified for nitrogen.

3.4.3. Compression ratio

Compression ratio for a certain gas is defined to be: $\mathbf{K = P_{outlet}/P_{intake}}$

$$\mathbf{K_{max}} = \left\{ \exp\left[\frac{V_B\sqrt{M}}{\left(\sqrt[2]{2k_B N_A T} \right)} \right] f_\alpha \right\}^n \quad (3.2)$$

"Vacuum Physics and Techniques" by T.A. Delchar.

Where M is the molar mass of the gas in question, V_B is the average tangential velocity of the blades, k_B is Boltzman's constant, N_A is Avogadro's number, T is the operating temperature, $f_{(\alpha)}$ is some function of the blade angle, and n is the total number of blades.

Maximum compression ratio depends exponentially on root of molar mass (M) and blade speed V_B.

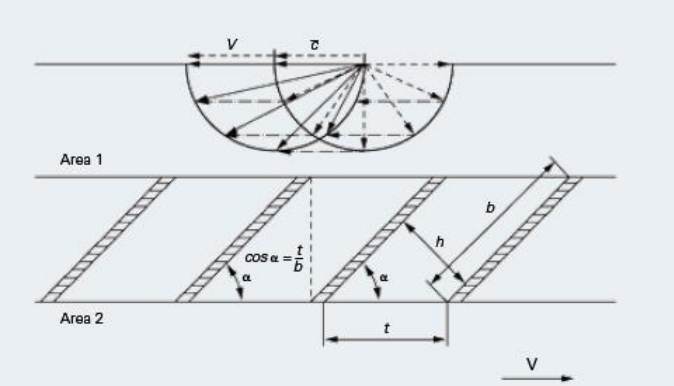

Fig 3.2.Blade Geometry

$$\mathbf{K_{max}} = exp\left[\frac{v}{\bar{c}*g*t*Sin\alpha}\right] \quad (3.3)$$

The factor *g* is between 1 and 3. From the relation, we can see that compression ratio increases exponentially by increasing blade velocity v and root of molecular mass M.

Mean thermal speed of molecules, $\bar{c} = \sqrt{\frac{8RT}{\pi M}}$ (3.4)

Since the molecular mass of N_2 is greater than O_2, compression ratio for nitrogen is significantly higher than Oxygen.

3.4.4. Pumping speed

Pumping speed of turbo-molecular pump S_0 has a relationship with the inlet area A and the blade speed or mean circumferential velocity of the blades *v*.

S_0= A * v *sin α * cosα, where α is blade angle (3.5)

But actual pumping speed will be somewhat less than the above one because of entry conductivity .If $L_{Bm} = \frac{\bar{c}}{4} * A$ is entry conductivity and the optimal blade angle is 45°, then the effective pumping speed S_{eff} of a turbo pump for gases whose molecular weight > 20 can be calculated as under:

$$S_{eff} = \frac{S_0 + L_{Bm}}{S_0 * L_{Bm}} = \frac{A.v}{4*\left[\frac{v}{\bar{c}}+1\right]} \quad (3.6)$$

3.4.5. Specific Pumping speed

The ratio between effective pumping speed and area of entry of the uppermost disk is known as specific pumping speed. But actual area may be reduced by a factor due to thickness of the blade. Therefore the relation becomes

$$S_A = \frac{S_{eff}}{A} = \frac{d_f.v\pi}{4*\left[\frac{v}{\bar{c}}+1\right]} \quad (3.7)$$

Where d_f is blade thickness factor ~= 0.9

Unit of specific pumping speed is l/s.cm^2.

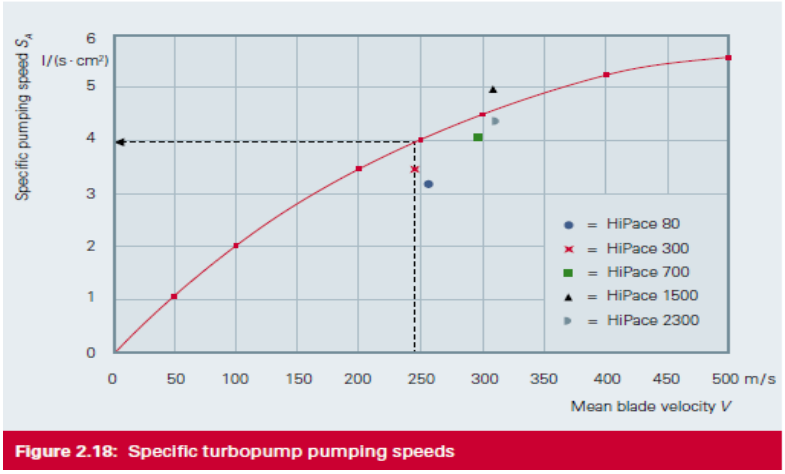

Figure 2.18: Specific turbopump pumping speeds

Fig 3.3.Specific Turbopump pumping speeds[4]

This is the plot between specific pumping speed and mean blade velocity. As every manufactures provide speed of the pump, we can calculate the mean blade velocity. Using eqn 3.5 effective pumping speed of can also be found out.

Mean thermal speed of nitrogen, $\bar{c}$ = 470 m/s

d_f = 0.9

from the plot we can calculate blade radii Ri and Ra also frequency of the pump.

Mean blade speed, $v = \pi f\,(Ra + Ri)$

Blade surface area, $A = \pi(R_a^2 - R_i^2)$

3.5. Evacuation of a chamber in the high vacuum region

Since the time required to reach desired high vacuum pressure depends greatly on outgassing from internal surface, the condition and pre-heating of these surfaces are of great importance in high vacuum and ultrahigh vacuum system. Porous regions exposed to vacuum may contribute enormous amount of outgassing. So the exposed surfaces must be very smooth and thoroughly cleaned. Gas evolution rate depends on the choice of material too and the surface condition.

The rate of gas evolution can be determined experimentally by the **pressure-rise method**: the system is evacuated first, then the chamber are isolated by valves. Now the time is recorded for the pressure to rise by a certain amount within the chamber of volume V. The gas quantity Q that arises per unit time is calculated from:

$$Q = \frac{\Delta p.V}{t} \qquad \Delta p \text{ is measured pressure rise} \tag{3.8}$$

If leak rate or gas evolution Q and the desired pressure p_{end} are known, we can determine the pumping speed by this formula:

$$S_{eff} = \frac{Q}{p_{end}} \tag{3.9}$$

3.6. Determination of a suitable backing pump

The gas or vapour quantity transported through a high vacuum pump must also be handled by the backing pump. Moreover, in the operation of the high vacuum pump (diffusion pump, turbomolecular pump), the maximum permissible backing pressure must never, even for a short time, be exceeded. If Q is the effective quantity of gas or vapour, which is pumped by the high vacuum pump with an effective pumping speed S_{eff} at an inlet pressure p_h, this gas quantity must certainly be transported by the backing pump at a pumping speed of SV at the backing pressure pV. For the effective throughput Q, the continuity equation applies:

$$Q = p_h \cdot S_{eff} = p_b \cdot S_b \tag{3.10}$$

The required pumping speed of the backing pump is calculated from:

$$S_b = \frac{p_h}{P_b} . S_{eff} \tag{3.11}$$

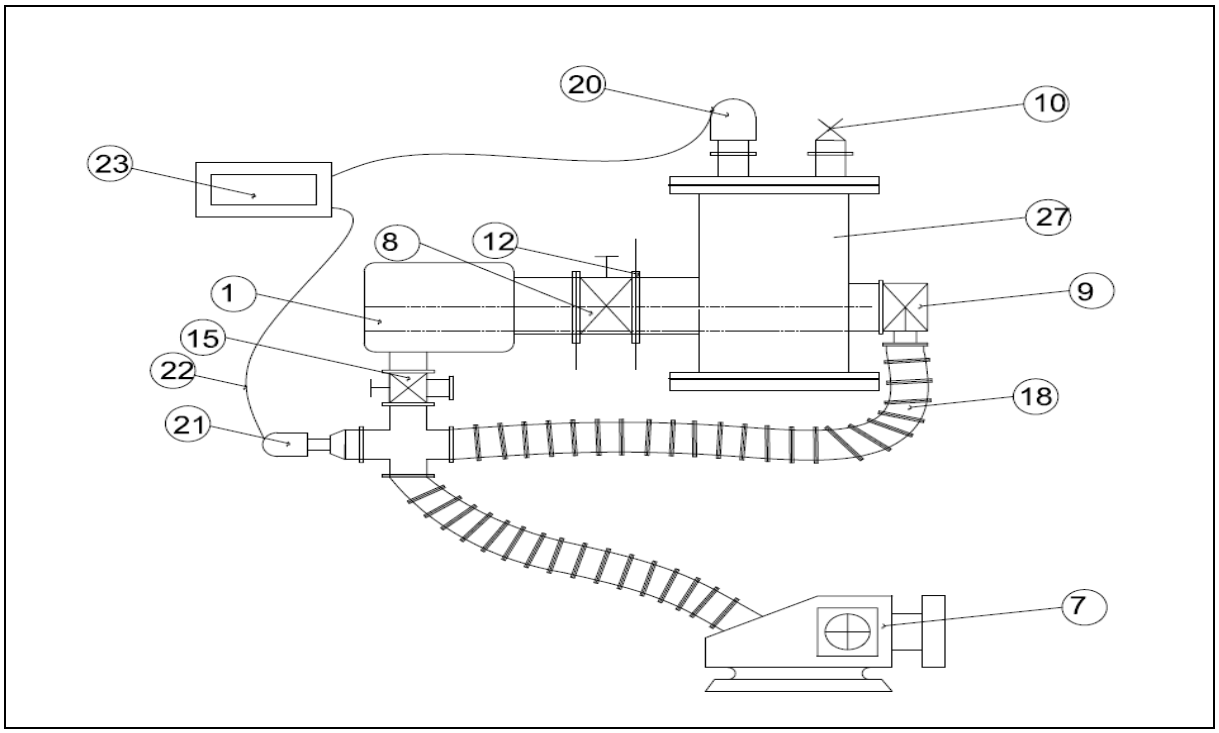

Fig 3.4. Turbomolecular Pump Experimental Set up

3.7. Item List-Turbomolecular Pump (Leybold)

S No	DESCRIPTION	PART NO	QTY
1	TURBOVAC 361 C; 100 ISO-K	85675	1
2	TurboDrive classic / RS 232	800075V0002	1
3	Connecting line NT 20; 3 m	85765	1
4	Mains cable, 3 m, Euro-plug	800102V0002	1
5	Air cooling unit TURBOVAC 151/361;230 V	85531	1
6	Purge /Vent valve,24 sccm, 100-230 V AC	800152V0014	1
7	TRIVAC D16B, 1-ph-Motor, 230 V, 50/60 Hz	11265	1
8	Gate Valve, DN 100 ISO-F H, Al, with p.g.f	28626	1
9	Right Angle Valve BAV 25 M SS	215385	1
10	Venting Valve, DN 10 KF, VA, Man	17337	2
11	Tee, DN 100 ISO-K VA	88736	1
12	Sealing Disk, DN 100 ISO-F Al, CR	17110	6
13	Centering Ring, DN 25 KF, VA,FPM	88347	25
14	Centering Ring, DN 16 KF, VA,FPM	88346	10
15	Straight-through Valve BIV 25 MSS	215374	1
16	T-PIECE DN 25 KF	88472	1
17	4-Way Reducer Cross, DN 25/16 KF VA	88496	1
18	Vacuum Hose DN 25 KF VA 1000mm	86803	2
19	Combi Transmitter PTR 90, DN 25 KF	230070	1
20	TM Transmitter TTR 91, DN 16 KF	230035	1
21	Connection Cable Type A, 5 m	12426	2
22	DISPLAY TWO(EU/US)	230024	1
23	Clamping Ring, DN 20/25 KF	18342	25
24	Clamping Ring, DN 10/16 KF	18341	5
25	Clamps, DN 63-250 ISO-K, 19-27 mm,(4 pc)	26701	4
26	Bolts, Nuts and Washers for DN 63-100 ISO F-Flange connection(set of 8 pc)	88781	2
27	Vacuum Vessel – 25 Ltr		1
28	Collar Flange with retaining ring, DN 100 ISO-F	26770	2
29	Reducer, DN 25/16 ISO-KF	88504	4
30	Blank Flange DN 25 ISO-KF	88437	10
31	Blank Flange DN 16 ISO-KF	88436	10

Table 3.1 Item List-Turbo-molecular Pump

3.8. Design

Volume of Vacuum Vessel = 25 Ltr

Assumed leak rate is <= $1*10^{-5}$ mbar l/s (Equipment is assumed to be sufficiently tight)

Also expected vacuum pressure, p_{end} = $<10^{-7}$ mbar.

Now leak rate and required pressure are known, the effective pumping speed can be calculated as;

$S_{eff} = \frac{Q_l}{P_{end}}, \rightarrow \frac{1*10^{-5}}{10^{-7}} \rightarrow S_{eff} = 100$ l/s, This S_{eff} is the minimum pumping speed required to pump out the assumed leak in gas to keep the system pressure 10^{-7} mbar.

If it required to reduce p_{end} further, two methods can be followed, either to reduce leak in rate or to increase S_{eff}.

∴ A higher capacity pump is selected with a pumping speed of 345 l/s (Nitrogen).

3.9. Technical details of Turbovac 361.

Technical Data		TURBOVAC 361	
Inlet flange	DN	100 ISO-K • 100 CF	160 ISO-K • 160 CF
Pumping speed			
N_2	l x s^{-1}	345	400
Ar	l x s^{-1}	350	–
He	l x s^{-1}	340	380
H_2	l x s^{-1}	340	370
Max. gas throughput			
N_2	mbar x l x s^{-1}	7.5	–
Ar	mbar x l x s^{-1}	7.5	–
Compression ratio			
N_2		1 x 10^9	1 x 10^9
He		6 x 10^4	6 x 10^4
H_2		3 x 10^3	3 x 10^3
Ultimate pressure	mbar (Torr)	< 1 x 10^{-10} (< 0.75 x 10^{-10})	< 1 x 10^{-10} (< 0.75 x 10^{-10})
Max. continuous inlet pressure [1]	mbar (Torr)	5 x 10^{-2} (3.75 x 10^{-2})	5 x 10^{-2} (3.75 x 10^{-2})
Max. foreline pressure for N_2	mbar (Torr)	5 x 10^{-1} (3.75 x 10^{-1})	5 x 10^{-1} (3.75 x 10^{-1})
Recommended forevacuum pump		from TRIVAC D 16 B to D 25 B	from TRIVAC D 16 B to D 25 B
Run-up time to 95% speed	min	≈ 2	≈ 2
Purge / vent port	DN	10 KF	10 KF
Cooling water connection (hose nozzle)	mm (in.)	10 (0.39)	10 (0.39)
Weight, approx.	kg (lbs)	12 (26)	12 (26)
Max. power consumption	VA	680	680
at ultimate pressure	VA	480	480

Table 3.2 Technical Data-TUBOVAC 361[8]

3.9.1. Maximum permissible Leak rate to get ultimate pressure

From the TURBOVAC 361 C catalogue it is seen that pumping speed of N2 as 345 l/s and ultimate pressure will be < 1 x 10^{-10}. Pumping speed of turbo pump does not change with pressure in its high vacuum region. Practically all pumping system may have some leakage and gas evolution. It cannot be avoided completely. But designers should try to avoid such

throughput as much as possible. The calculation below shows the permissible leak rate to obtain ultimate pressure of the pump.

Leak rate, Q= Seff/ Pend,

In this design turbo pump is directly connected to vacuum chamber with no vacuum fittings in between. Therefore we will get maximum pumping speed at chamber without any loss.

So effective pumping speed S_{eff} = 345 l/s (N). Taken from the table.

Q = 345/ 1 x 10^{-10}, → Q = 3.45*10^{-8} mbar l/s which is < 10^{-6} mbar .l/s.

In order to obtain 1 x 10^{-10} mbar by this vacuum system, the equipment should be very tight. If any unavoidable chance of more leak than the allowable rate, either a higher capacity (pumping speed) must be chosen or to fit additional pump to cop up with the extra leak to get the ultimate vacuum pressure of 1 x 10^{-10} mbar. Otherwise we should compromise with the ultimate pressure which the existing system can create.

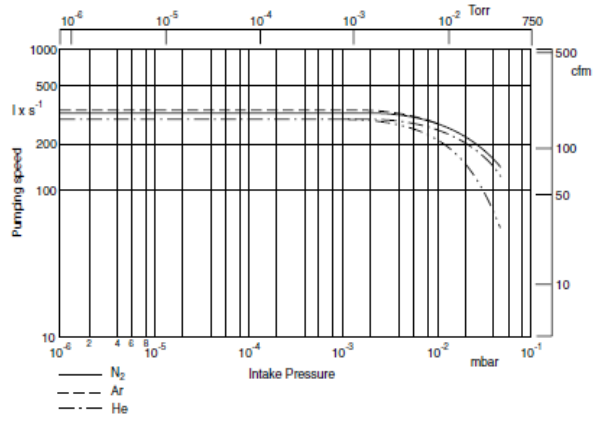

Fig 3.5. Pumping Speed Characteristics- Turbo Pump

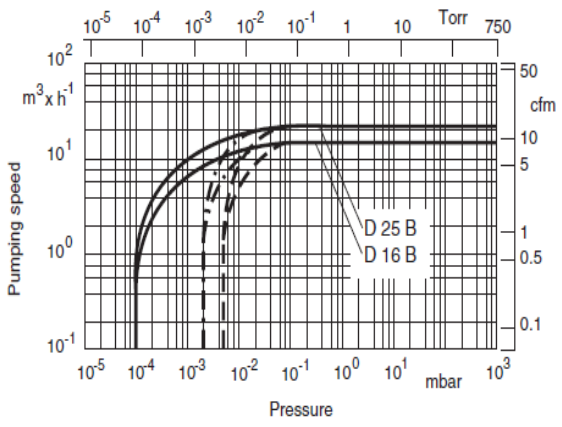

Fig 3.6. Pumping Speed Characteristics-Rotary Pump

3.9.2. Selection of Backing Pump

Inlet pressure of turbo pump for constant pumping speed = 5x 10^{-3} mbar

Maximum permissible backing pressure = 5x 10^{-1} mbar

Pumping speed of backing pump, $S_b = \frac{5*10^{-3}}{5*10^{-1}}.345 = 12.42$ m³/h. $S_b = \frac{p_h}{P_b}.S_{eff}$

Suitable rotary pump in this range is one with a pumping speed of 16 m^3/h (Leybold).

∴ TRIVAC D16B Rotary pump is selected.

3.9.3. Experiment Procedure:

1. Open the isolating valve of turbo-molecular pump
2. Start the double stage rotary backing pump and simultaneously start counting time using a stop watch.
3. Take the reading of vacuum gauge after each particular interval of time.
4. Allow the roughing/backing pump to create a vacuum in the order about 10^{-3} mbar.
5. Then start the turbo-molecular pump.
6. Note down the vacuum gauge reading till it reaches its ultimate vacuum pressure.
7. On reaching the designed ultimate pressure, to stop the system, first turn off the turbo-molecular pump.
8. Allow the rotary backing pump to rotate for further half an hour.
9. Close the isolation valve. Switch off the rotary pump.
10. Tabulate the readings. Calculate pumping speed for different pressure ranges and the plot graphs.

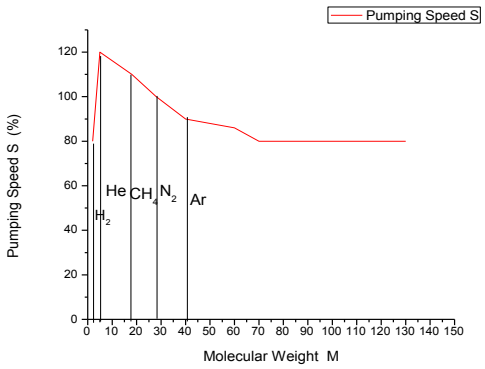

Fig.3.7 Pumping Speed Varies with Molecular Weight

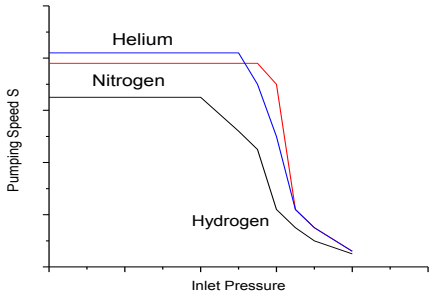

Fig.3.8 Pumping Speed Vs Inlet Pressure

CHAPTER 4

SORPTION PUMP

Aim

To design and construct sorption pumping station and prepare the experimental procedure to conduct the experiment.

4.1. Introduction

The term “sorption pumps” means the removal of gases and vapours from a space to be evacuated by the process of adsorption. The pumped gas particles are getting attached with the surfaces or in the interior of these agents, due to either by physical temperature-dependent adsorption forces (van der Waals forces), chemisorption, absorption, or by becoming embedded during the course of the continuous formation of new zorbing surfaces.

Sorption pumps provide us a safe, clean, quiet, vibration free and inexpensive way to rough pump a vacuum system up to 10^{-3} mbar. They are generally used where the vacuum systems are sensitive to oil contamination from normal mechanical pumps. All sorption pumps work by **gas-capture** process. With prolonged use, the material become "saturated" and may reduce gas-capture ability. For enhancing or to retain its capacity further a sorption pump will either need to be "regenerated" or replaced.

4.2 Principle of Operation

Sorption roughing pumps or sorption pumps are used for pumping systems from atmospheric pressure to a pressure of approximately 10^{-2} mbar. They rely on the binding forces existing between a gas and a surface or gas and adsorbent material. In other words, they pump by cryosorption. A typical Sorption pumps consist of a cylindrical body (canister) filled with an adsorbent material. The adsorbent is usually a molecular sieve material, or zeolite (activated Al2O3), which consists of pellets made of calcium or a sodium alumino-silicate crystalline matrix. The canister is surrounded by a dewar. Annular space is filled with liquid nitrogen. Since Zeolite has poor heat transfer properties, an array of aluminium fins are provided inside the pump to improve thermal contact with the sieve material. The pump body as well as internal cooling fins are carefully designed to maximize heat transfer rate. To reduce axial heat transfer outside the pump body, neck and flange are made out of stainless steel.

Sorption pumps are non-contaminating, clean and vibration free roughing pumps and are ideal for low throughput applications. They are used as backing pump for getter pumps, ion pumps, or mechanical cryopumps due to its oil free working. In a sorption pump, molecules are captured on the zeolite surface by physical adsorption. The number of molecules that can be held on an adsorbent is dependent on the temperature of both gas and surface, the chemical nature of gas and surface, the microscopic roughness of the surface, and the incident flux of molecules.

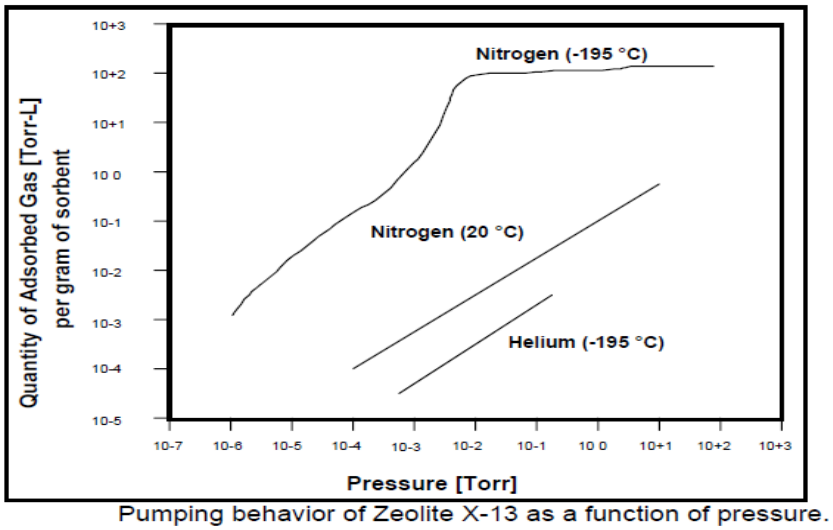

Figure 4.1 Pumping Behaviour of Zeolite as a function of Pressure [1]

Zeolite Sorption pump provide a very large surface area which is kept under a temperature below the boiling point of most constituent gas present in the place where we need to create a vacuum atmosphere. Gas molecules being pumped come in contact with the surface and get attached and physically captured.

The adsorbent, which made of zeolite 13X has a surface area to mass ratio (about 103 m^2 per gram) and diameter of about 13Å ($1.3*10^{-9}$ m). This is approximately the size of a molecule of water, oil vapour and larger gas molecules (nitrogen and oxygen). This pore size is sufficient to capture molecules of Nitrogen and Hydrogen present dominantly in the atmosphere. But low atomic weight gases having molecular diameters smaller than the 13Å are captured less effectively by zeolite. Absorption of gases by a given sorbent depends mainly on gas specie, temperature of sorbent surface, and gas pressure. As nitrogen gas is cooled, the amount of gas that can be adsorbed by the zeolite per gram increases.

4.3. Design

Vacuum Chamber volume = 5 Ltr

Pumping Capacity of Zeolite material = 30 Torr-litres of air /gm (cooled to 77 K)

$\therefore$ Mass of zeolite required = $\frac{\text{Chamber Volume* Atmospheric pressure}}{\text{Pumping Capacity per gm}} \rightarrow \frac{5*760}{30}$

= 126 gm

To evacuate 5 Ltr chamber at least 126 gm zeolite is to be taken in the pump. But this much amount of zeolite may get saturated after single use. By considering repeated use in single experiment without regeneration, we take 5 times of the actual amount required for single use.

Total mass of Zeolite = 126* 5= 630 gm, and $\rho_{Zeolite}$ = 1.33 gm/cc.

$\therefore$ Total pump volume occupied by Zeolite = $\frac{\text{Total mass of Zeolite}}{\text{Density}} \rightarrow \frac{630}{1.33}$ = 473 cc.

Approximate 0.5 Ltr of pump volume may occupied by Zeolite itself. Volume of aluminium fins should also be taken into account. So we select a 3 Ltr Aluminium chamber for pump having wall thickness of 2mm.

Vacuum Chamber Volume	5 Ltr
Pump Volume	3 Ltr each
Zeolite	650 gm each

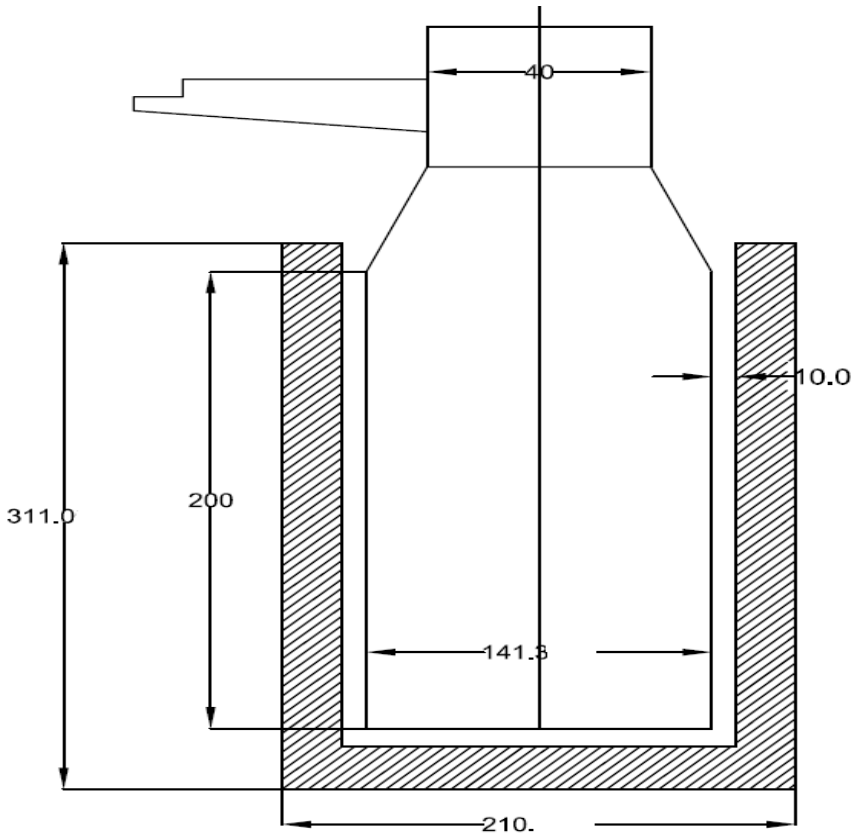

Fig 4.2 Sorption Pump Details

4.4. Construction and Working

The main parts of sorption pump consists of stainless steel body and an array of fins that remove heat from the zeolite. A pressure relief mechanism is fitted with an elastomer stopper to releases positive pressure when exceeds the limit.

The adsorbent used is zeolite 13 A which is highly porous with a surface to volume ratio of about 103 square meters per cubic centimetre. These pores are large enough to trap nitrogen, oxygen, and argon molecules, the main constituents of air. Zeolite also possess a very high affinity for water vapour. Water vapour accumulated through repeated pumping process may saturate the sieve material, reducing and eliminating its capacity for adsorbing nitrogen and oxygen. Periodical heating up to 250°C is necessary to remove the accumulated water vapour and regenerate the adsorbent material.

During pump operation, do not turn on the heater since it is cooled by liquid nitrogen. Pumping of neon and helium along with other air constituents is very difficult. And if neon is pumped together with air, its capacity will be less because the neon will be replaced by the active air

gases, starting at pressures below 7.5 mbar. For eliminating this difficulty, sorption pumps are used in multistage. If two pumps are staged, first pump is used to bring down the pressure up to 7.5 mbar and is then shut it off. The second stage pump is then opened and the pressure is further reduced. About 99% of the air can be removed with the first pump, and noble gases which are already swept into this pump cannot back stream into the system again when pressure is further reduced while second stage pump is in operation. These pumps are often fitted with both Bourdon and thermocouple vacuum gauge for monitoring vacuum levels. Isolation of one stage from another is done with the help of angle valves. Entire pumping operation is fast and simple. Before starting we need to pour liquid nitrogen to the dewars up to a certain level. As soon as the adsorbent material in the pump is chilled with liquid nitrogen pump down will start. A single stage sorption pump can evacuate a 100 litre chamber from atmosphere to 10^{-2} mbar in approximately within 10 minutes. Multiple pump systems are commonly used as they are faster and more efficient.

4.5. Experimental Setup-Sorption Pump

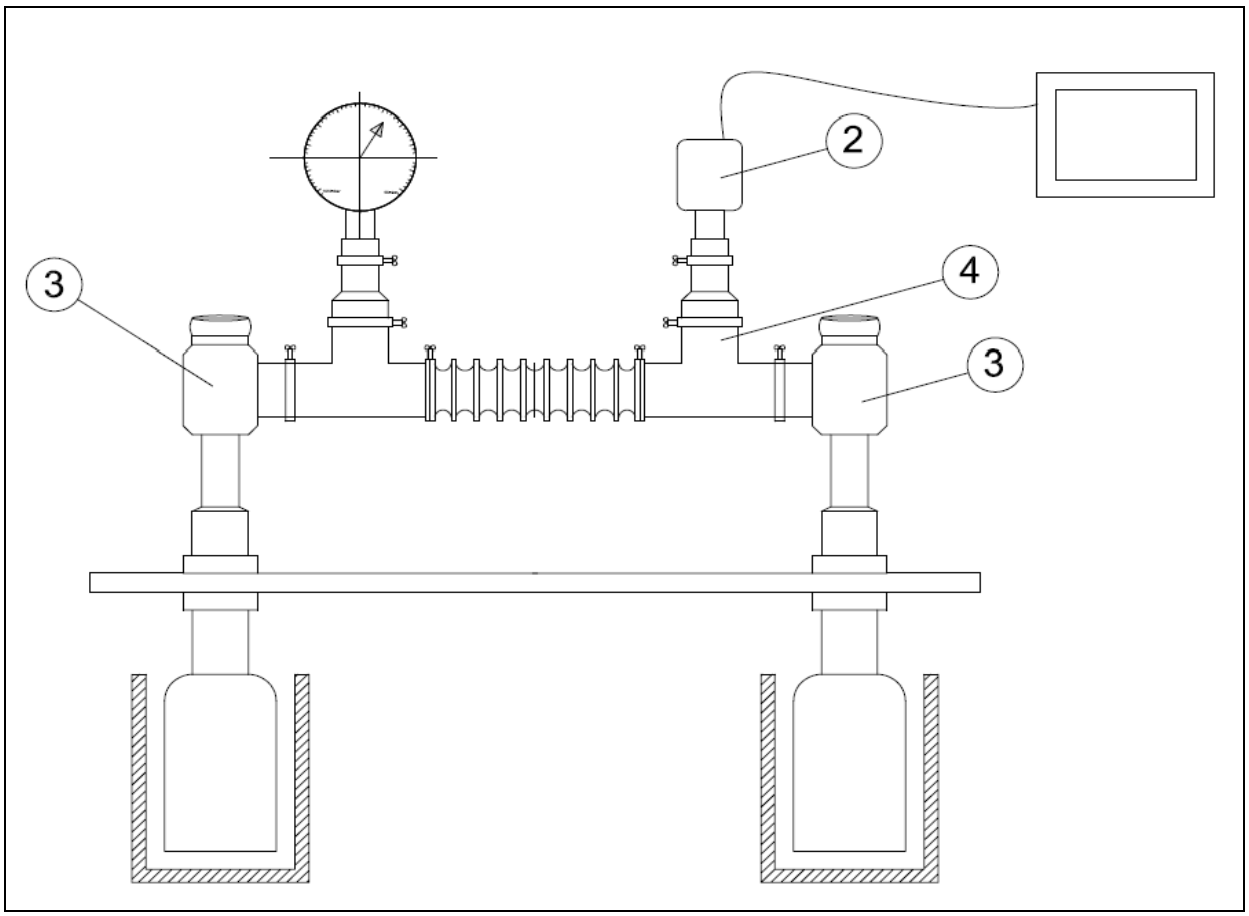

Fig 4.3 Sorption Pump Experimental Setup

4.6. Item List- Sorption Pump

S.No	DESCRIPTION	SPECIFICATION	QTY
1	Vacuum Vessel(Cylindrical with top Lid)	5 Ltr, Ports-1X DN 25 ISO-KF Side or on Top	1
2	Thermocouple Gauge		1
3	Right Angle Valve(Manual)	DN 25 ISO-KF	2
4	Tee	DN 25 ISO-KF	1
5	Elbow	DN25KF(Radial) 90 degree	1
6	Bourden Tube Vacuum Gauge(1013-50 mbar)		1
7	Hinged Clamp	25 KF	9
8	Centering Ring	DN 25 ISO-KF	9
9	Sorbent Material	SPMS-150(MDC Vacuum)	1
10	Stopper/Pressure Relief Components - Pop-off Pressure Relief	Pressure relief valve	2

Table 4.1 Item List –Sorption Pump

4.7. Experimental Procedure:

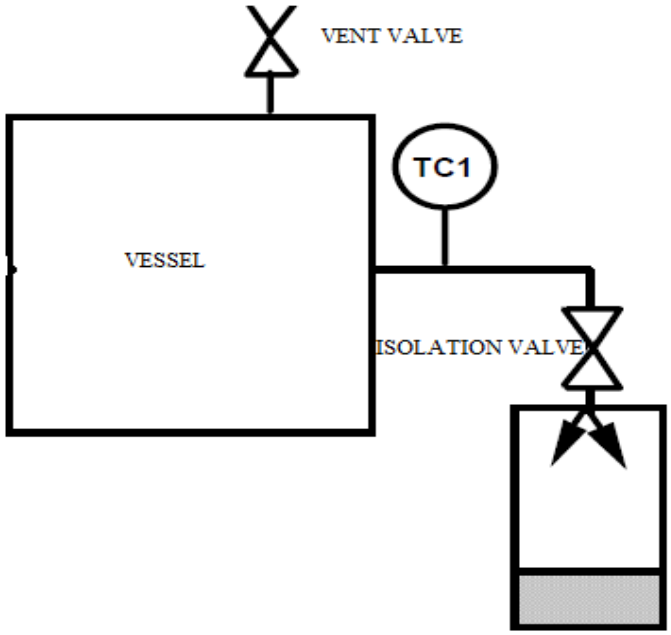

Fig 4.4 Line diagram of Sorption Pump

1. If regeneration is required, bake the pump up to 250^0 C or higher.
2. Allow it to cool down to room temperature with the relief valve and isolation valve closed.
3. Fill the Polystyrene dewar sufficiently with Liquid Nitrogen.
4. Allow 20- 30 min for the sorbent to cool.
5. Isolate vacuum chamber by closing vent valve.
6. With the vessel at ambient condition, open isolation valve and simultaneously start recording pressure along with time till the system reaches its ultimate pressure.
7. Close isolation valve and vent off the vessel.
8. Again close the vent valve and repeat the experiment.

CHAPTER 5

LEAK DETECTION

Aim

This exercise will give student an introduction to the leak detection in vacuum systems. Familiarisation of various methods to detect leaks as well as the most widely used helium leak detectors and its different applications are to be taught. In addition, the method of using a Helium Mass Spectrometer Leak Detector (HMSLD) to locate vacuum system leaks is thoroughly covered. Provide them practical knowledge to properly characterize, operate, and maintain vacuum system for maximum uptime with avoidable leak rate.

5.1. Introduction

Normally a vacuum chamber should maintain the achieved vacuum pressure forever after isolating and switching off the pumps. However in most of the cases without active pumping the pressure will rise with time. The pressure rise may be either by outgassing; evolution of gas molecules from chamber walls; or by gas molecules penetrating through leaks and entering by permeation from the outside into the vacuum system.

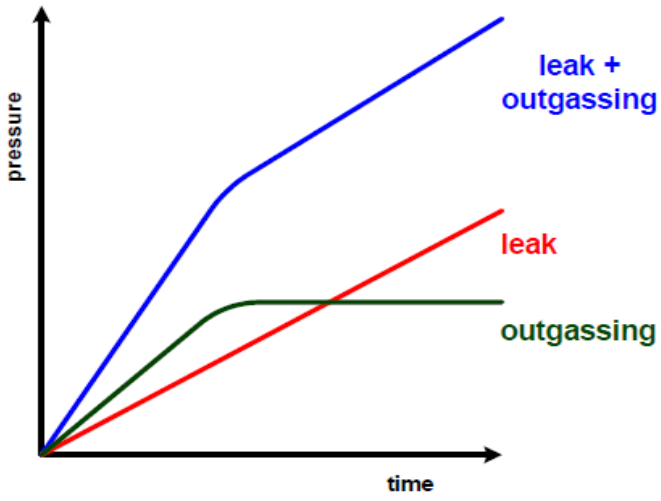

Fig 5.1 Variation of Pressure in an isolated system

Practically all the vacuum system have some amount of leak. Perfect leak proof system is impossible to build and not necessary. But the leak rate should be small enough so that we can attain the required pressure level and maintain it for considerable time. Construction of vacuum vessel should follow some specific standard welding and testing procedure to get the tightness of the vessel within allowable range. During installation as well as after assembly leak detection

checks are necessary to locate possible leak. Therefore leak detection is an important procedure to be followed to reach required pressure level and also to maintain the vacuum level.

5.2. Leak Rate

The throughput of a gas in pressure Volume term i.e. mbar l/s through a leak is known as leak rate. It depends on type of gas, pressure differential across the leak and temperature of gas. A system having volume *V* and a pressure rise Δp within a time interval Δt; then leak rate Q_l is given by,

$$Q_l = \mathrm{V} * \frac{\triangle p}{\triangle t} \tag{5.1}$$

When type of gas, mass flow and temperature are given, then we can find out volume flow rate through the leak by using;

$$Q_l = \frac{\triangle (p.V)}{\triangle t} = \frac{R.T}{M} . \frac{\triangle m}{\triangle t} \tag{5.2}$$

Where R = 83.14 mbar. l/mol. K, T=temperature in K; M=molar mass in g/mole; $\frac{\triangle m}{\triangle t}$ is the loss of mass in t seconds through the leak

Thumb rule for leak rate for high vacuum application.

Leak rate < 10^{-6} mbar .l/s	Equipment is very tight
Leak rate 10^{-5} mbar .l/s	Equipment is sufficiently tight
Leak rate > 10^{-4} mbar .l/s	Equipment is leaky

The effect of can be overcome by installing with sufficient pumping speed; lowest pressure that can be attained in a system having leak rate Q_l by a pump of capacity S_{eff} is given by;

$$P_{end} = \frac{Q_l}{S_{eff}} \tag{5.3}$$

5.3. Sources of leaks

- Leaks from temporary connections; Flanges, seals, covers, centering rings etc
- Leaks from permanent connections; Welding and soldering joints.
- Leaks from porous walls.
- Gas liberation leaks.
- Permeation of outside gas through sealing materials, rubber hoses.

5.6. Leak Detection Methods

Leak detection tests are done either to locate the exact place of leak or to determine the total rate of leak. Different methods are used based on the variation of physical property within the vacuum system while the system being kept at pressurised or evacuated condition.

Methods based on Mechanical effects

- Ultra sound detectors- Leak rate up to 10^{-2} mbar. l/s
- Soap bubble test- Leak rate up to 10^{-4} mbar. l/s

The above methods are cheap, simple and easy to carry out. Since the sensitivity is low, it can be useful up to high vacuum region. UHV

5.6.1. Pressure rise method

If the system having some leak, there will be a continuous rise of pressure linearly with time. But sometimes outgassing also cause to increase pressure, but outgassing pressure rise may come to a standstill condition after some duration of time. By analysing the Pressure Vs time curve time we can predict whether a leak is exist or not. But still we cannot locate the leak.

5.6.2. Tracer gas method

Local spraying of tracer gas on vacuum chamber may change the composition of residual gas remaining inside the chamber. By measuring the physical properties of residual gas we can determine the locality and size of leak. Mass spectrometer analysis is the most sensitive and popular method in leak detection. With helium as tracer gas, using a mass spectrometer leak rate down to 10^{-12} mbar. l/s can be measured.

5.7. Mass Spectroscopic Leak detection

5.7.1. Test methods

- Local leak detection- Locate the spot of leakage
- Total leak detection- Determination of total leak rate.

5.7.2. Operating methods:

Pump-down method- Helium is sprayed over the evacuated chamber.

Sniffer method- Chamber is filled with a helium overpressure Δp of more than 100 mbar. Leaked helium from the chamber which is being collected by an outer envelope is sucked into the leak detector via a sniffer.

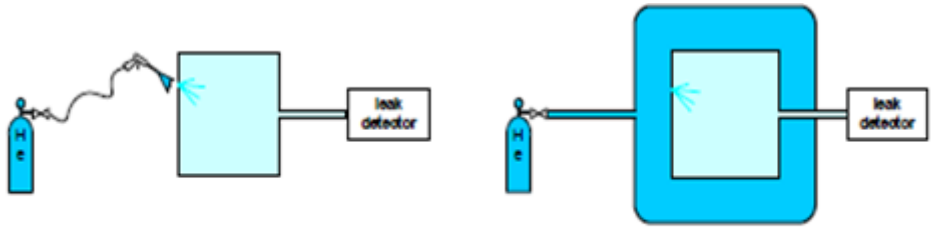

Fig 5.2 Leak detection of vacuum vessels using the tracer method – left: leak location, right: total leakage measurement.

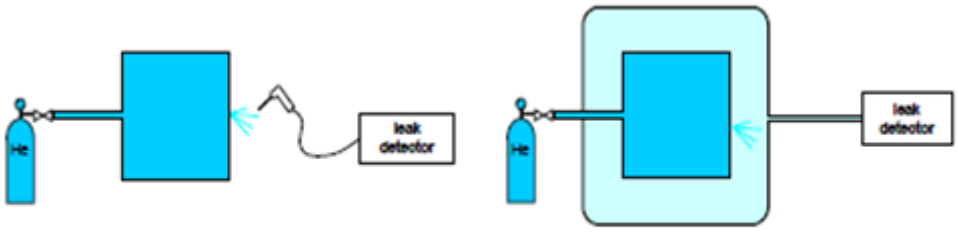

Fig 5.3 Leak detection of pressure vessels using the detector method – left: leak location using a sniffer, right: total leakage measurement.

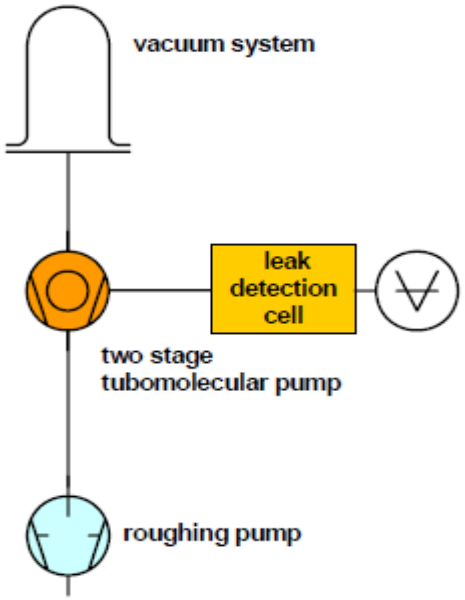

Fig 5.4 Leak Detection Counter Flow method

5.8. Working of Helium Leak Detection Cell

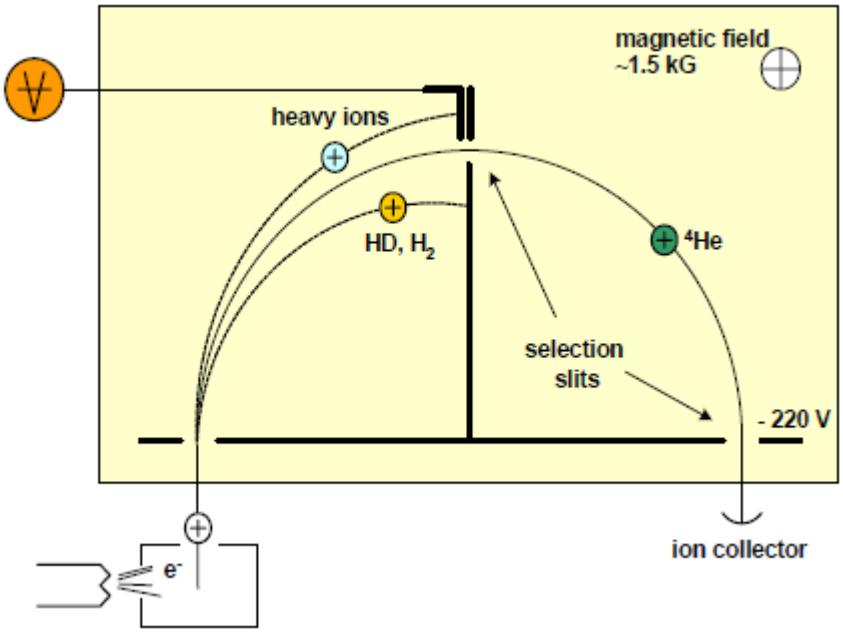

Fig 5.5 Working of He Leak Detection

Main parts of the cell are ion source, separation system and ion collector. The residual gas being pumped from the chamber is ionized by electron bombardment. The ionized gas is accelerated by a magnetic field, deflected and finally only helium atoms are allowed to pass

through the slit provided in the middle of magnetic field. An ion collector which is placed outside the magnetic field collects 180°deflected helium ions, which is then converted in to partial pressure value of helium. All other atoms are blocked inside the magnetic system. Second collector placed at the middle is used to measure heavy ion current flow, and then it is converted into a total pressure value.

At its highest sensitivity of 10^{-12} mbar· l/s, a corresponding current of 10^{-15} A must be measured. Electron multipliers helps in such extreme measurement in the most modern detectors.

CHAPTER 6

RESULT & CONCLUSION

6.1. Result & Conclusion

Design, construction and installation of Rotary pump experimental set up are completed. Pumping Speed measurement of rotary pump is carried out on the equipment.

Pressure vs time graph and pumping speed characteristics of rotary pump by constant volume method are obtained.

Design and drawings of Turbo-molecular pump and Zeolite Sorption pump have been done. Bill of material for both the experiments are prepared.

Leak detection experiment is already available in the lab. Once the turbo-molecular pumping station is installed, leak detection test can also be done with the existing device.

6.2. References

[1] Las Positas College, Vacuum Technology 60A & 60B

[2] V.V. Rao - Vacuum Science and Technology

[3] C. Hauviller, Design rules for vacuum chambers, CERN, Geneva, Switzerland

[4] Pfeiffer Vacuum: Working with Turbo pumps: Introduction to high and ultrahigh vacuum production. http://www.pfeiffer-vacuum.com. (2003).

[5] K. Zapfe, Technical Note on Leak Detection, (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany 2007)

[6] M.Hablanian, Marsbed, Cryo-sorption pumps-High Vacuum Technology, Marcel Dekker, INC, New York, New York. 1990.

[7] David Garton,Vacuum Technology and Vacuum Design Handbook for Accelerator Technicians, November 2011, Revision 0

[8] Fundamentals of Vacuum Technology, Leybold Vacuum, 00.200.02 Kat.-Nr. 199 90